# C-MAC® Test kit

# User's Manual





ITEM	METHOD	PAGE
Aluminum	Aluminon	. 1
Bromine	DPD	<u> </u>
Chlorine, Free	DPD	=
Chlorine, Total	DPD	. <b>7</b>
Chlorine Dioxide	DPD	
Chrome, Hexavalent	1,5 Diphenylcarbohydrazide	. 11
Chrome, Total	Alkaline Hypobromite Oxidation	
Copper	Bicinchoninate	. 17
Copper	Porphyrin	. 19
Cyanide	Pyridine - Pyrazalone	. <b>21</b>
Cyanuric Acid	Turbidimetric	. <b>25</b>
Fluoride	SPADNS	. <b>27</b>
Hardness	Calmagite Colorimetric	. 29
Iron	Iron	. 33
Iron, Ferrous	1,10 Phenanthroline-25mL	. <i>35</i>
Iron, Total	1,10 Phenanthroline-10mL	. <b>37</b>
Manganese, LR	PAN	. <b>39</b>
Manganese, HR	Periodate Oxidation	. <b>41</b>
Nitrate, HR	Chromotropic Acid	. 43



ITEM	METHOD	PAGE
Nitrate, LR	Cadmium Reduction	<b>45</b>
Nitrate, MR	Cadmium Reduction	47
Nitrate, HR	Cadmium Reduction	. <b>49</b>
Nitrite, LR	Diazotization	<b>51</b>
Nitrite, LR	Diazotization; Test kit	<b>53</b>
Nitrite, HR	Ferrous Sulfate	. <b>55</b>
Nitrogen, Ammonia, LR	Salicylate	<b>57</b>
Nitrogen, Ammonia, HR	Salicylate	<b>59</b>
Nitrogen, Total: TN, LR	Acid Persulfate	<b>61</b>
Nitrogen, Total: TN, HR	Acid Persulfate	<b>65</b>
Oxygen Demand, Chemical: COD <sub>Cr</sub> , ULR	Reactor Digestion	<b>69</b>
Oxygen Demand, Chemical: COD <sub>Cr</sub> , LR	Reactor Digestion	<b>71</b>
Oxygen Demand, Chemical: COD <sub>Cr</sub> , HR	Reactor Digestion	<b>73</b>
Oxygen Demand, Chemical: COD <sub>Cr</sub> , UHR	Reactor Digestion	<b>75</b>
Phosphorus,Reactive :Orthophosphate, LR	Acid Persulfate	77
Phosphorus,Reactive :Orthophosphate, HR	Molybdovanadate	. <b>79</b>
Phosphorus, Total: TP, LR	Acid Persulfate	<b>81</b>
Phosphorus, Total: TP, HR	Molybdovanadate	· <i>85</i>
Silica	Silicomolybdate	<b>89</b>
Sulfate	Sulfate	. <b>91</b>
Sulfide	Methylene Blue	. <b>93</b>
Zinc	Zincon	<b>95</b>



# Aluminum (0.008 $\sim$ 0.800 mg/L ${\rm Al}^{3+}$ )

# Aluminon Method

Required Reagents	Aluminum Reag Ascrobic Acid F Bleaching Reag	Pillow	Cat. NO.	10810-00		
	Acidity	If greater than 300 mg/L acidity as CaCO <sub>3</sub> , Add one drop of m-Nitrophenol Indicator S and 5N NaOH Solution to the sample. Invert to mix. Repeat as often as necessary und color changes from colorless to yellow. Add one drop of 5.25 N Sulfuric Acid Standar Solution to change the solution from yellow back to colorless.				
Interferences	Alkalinity	1000 mg/L as CaCO <sub>3</sub> : Add one drop of m-Nitrophenol Indicator Solution to the sample. A yellow color indicates excessive alkalinity. Add one drop of 5.25 N Sulfuric Acid Standard Solution to change the solution from yellow back to colorless.				
	Fluoride	At all leves				
	Iron	Greater than 20 mg/L				
	Phosphate	Greater than 50 mg/L				
	Polyphosphate	At all levels by causing negative e	errors. Must be c	converted to orthophosphate.		
Sampling Storage & Preservation	Collect samples in a clean glass or plastic container. Preserved the sample by adjusting the pH to 2 or less with nitric acid(about 1.5mL per liter). Can be 6 months at room temperature. Before analysis, adjust the pH to 3.5 ~ 4.5 with 5.0 N NaOH solution.					
Tips & Techniques	Digestion is required for determining total aluminum. Clean glassware with 6.0 N HCl and Deionized water before analysis. The sample temperature must be between 20 ~ 25 for accurate results. Clean glassware with soap and a brush immediately following analysis.					

**Aluminum** 

## 1

# Procedures Aluminon Method



1. Fill the cylinder to the **50 mL** and Add the contents of one Ascorbic Acid Pillow. Stopper. Invert several times to dissolve powder.



2. Add the contents of one Aluminum
Reagent Powder Pillow. Stopper. Invert for
1minute to dissolve the powder completely.
(Red-Orange color will develop if Aluminum is present)



**3.** Pour **25 mL** of the mixture into a 25 mL sample cell.(This is the prepared sample.)



**4.** Add the contents of one Bleaching Reagent Pillow to the remaining 25mL in the cylinder. Stopper. Shake for **30 seconds** vigorously.



5. Pour the 25mL of solution from the cylinder into a second 25mL sample cell.A 15 minute reaction period will begin.(This is the blank.)



**6.** After choosing C-MAC mode in the program, choose **Prog.# 1.** 

(HACH DR/890 : 1

DR/2010 & 2500 : 10

DR/4000 : 1000)



7. Within 3 minutes after the timer beep, wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**8.** Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

Press Enter.

(Results will appear in mg/L Al3+)



# Bromine $(0.05\sim4.50 \text{ mg/L Br}_2)$

# DPD Method

Required Reagents	DPD Free Chlorine Reagen	t Pillow	Cat. NO.	11010-00		
	Acidity	Greater than 150	mg/L as CaCo	O₃ : Neutralize with 1N NaOH		
	Alkalinity	Greater than 250	mg/L as CaC(	O₃ : Neutralize with 1N H₂SO₄		
	Chlorine, Chlorine Dioxide	At all levels				
	Chloramines, organic	May interfere				
	Hardness	No effect at less	than 1,000 mg	g/L as CaCO₃		
	lodine	At all levels				
Interferences	Mn <sup>4+</sup> ,Mn <sup>7+</sup> or Cr <sup>6+</sup>	After adjusting sample pH to 6-7, add 3 drops KI(30g/L) to a 25mL sample. Mix and wait 1 minute. Add 3 drops sodium arsenite(5g/L) and mix. Analyze 10mL of the treated sample. Substract the result from this test from the original analysis to obtain the correct bromine concentration.				
	Monochloramine, Ozone	At all levels				
	Peroxides	May interfere				
	Extreme sample pH or highly buffered samples	Neutralize to pH 6~7				
Sampling Storage & Preservation	Collect samples in clean, dry glass containers. If sampling from a tap, allow the water to flow at least 5minutes to ensure a representative sample. Avoid excessive agitation and exposure to sunlight.  Allow several volumes of water to overflow the container and cap the container so there is no headspace above the sample. Analyze samples immediately. Do not preserve.					
Tips & Techniques	If the samples temporarily turns yellow after reagent addition, dilute a fresh sample and repeat the test. For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample Subtract the reagent blank value from the final results of perform a reagent blank adjust.					

Bromine 3

# 1

## Procedures DPD Method



1. Fill a sample cell with 10 mL of sample.



2. Add the contents of one DPD Free Chlorine Reagent Pillow to the sample cell (the prepared sample). Stopper. Invert to dissolve the powder. A 3-minute reaction period will begin. A pink color will develop if Bromine is present.



**3.** Fill a second sample cell with **10 mL** of sample.(the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 4**.

(HACH DR/890 : 4

DR/2010 & 2500 : 50

DR/4000 : 1300)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



6. Within 3 minutes after the timer beep, wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Br<sub>2</sub>)

Bromine



# Chlorine, Free (0.02 $\sim$ 2.00 mg/L Cl<sub>2</sub> )

# DPD Method

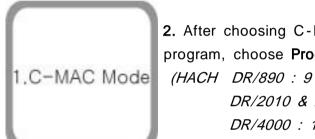
Required Reagents	DPD Free Chlorine Reagent Pil	low	Cat. NO.	11210-00		
	Acidity	Greater than 150 mg/L as CaCO <sub>3</sub> : Neutralize with 1N NaOH				
	Alkalinity	Greater than 250 mg/L as CaCO <sub>3</sub> : Neutralize with 1N H <sub>2</sub> SO <sub>4</sub>				
	Bromine, Br <sub>2</sub> , Chlorine Dioxide	At all levels	3			
	Chloramines, organic	May interfe	re			
	Hardness	No effect a	at less than 1,000	mg/L as CaCO <sub>3</sub>		
	lodine, I <sub>2</sub>	At all levels	3			
Interferences	After adjusting sample pH to 6-7, add 3 drops KI(30g/L) to Mix and wait 1 minute. Add 3 drops sodium arsenite(5g/L)  Analyze 10mL of the treated sample. Substract the result from the original analysis to obtain the correct bromine con					
	Monochloramine	When read within 1 minutes after reagent addition, 3mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.				
	Ozone	At all levels				
	Peroxides	May interfe	re			
	Extreme sample pH or highly buffered samples	Neutralize to pH 6~7 with 1N H <sub>2</sub> SO <sub>4</sub> or 1N NaOH				
Sampling Storage & Preservation	dilute bleach solution (Bleach water. If sampling from a tap,	1mL per lite allow the w to overflow	r) for at least 1ho ater to flow at lea the container an	o remove any chlorine demand by soaking in a bur. Rinse thoroughly with deionized or distilled ast 5minutes to ensure a representative sample. It is no headspace eserve.		

Chlorine, Free

#### **Procedures DPD** Method



1. Fill a sample cell with 10 mL of sample. (the blank)



2. After choosing C-MAC mode in the program, choose Prog.# 9.

DR/2010 & 2500 : 80

DR/4000 : 1450)



3. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



4. Fill a second sample cell with 10 mL of sample.



5. Add the contents of one DPD Free Chlorine Reagent Pillow to the sample cell. (the prepared sample). Swirl the sample cell for 20 seconds to mix.



6. Within 1 minute of adding the reagent, wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter. (Results will appear in mg/L Cl<sub>2</sub>)

Chlorine, Free



# Chlorine, Total (0.02 $\sim$ 2.00 mg/L Cl<sub>2</sub>)

# DPD Method

Required Reagents	DPD Total Chlorine Reagent Pi	llow	Cat. NO.	11310-00		
	Acidity Alkalinity Bromine, Br <sub>2</sub> , Chlorine Dioxide Chloramines, organic Hardness Iodine, I <sub>2</sub>	Greater than 150 mg/L as CaCO <sub>3</sub> : Neutralize with 1N NaOH Greater than 250 mg/L as CaCO <sub>3</sub> : Neutralize with 1N H <sub>2</sub> SO <sub>4</sub> At all levels May interfere No effect at less than 1,000 mg/L as CaCO <sub>3</sub> At all levels After adjusting sample pH to 6-7, add 3 drops KI(30g/L) to a 25mL sample				
Interferences	Mn <sup>4+</sup> ,Mn <sup>7+</sup> or Cr <sup>6+</sup>	Mix and wait 1 minute. Add 3 drops sodium arsenite(5g/L) and mix.  Analyze 10mL of the treated sample. Substract the result from this test from the original analysis to obtain the correct bromine concentration.				
	Monochloramine			after reagent addition, 3mg/L monochloramine increase in the reading.		
	Ozone	At all level	S			
	Peroxides	May interfe	ere			
	Extreme sample pH or highly buffered samples	Neutralize to pH 6 ~ 7 with 1N H <sub>2</sub> SO <sub>4</sub> or 1N NaOH				
Sampling Storage & Preservation	dilute bleach solution (Bleach 1mL per liter) for at least 1hour. Rinse thoroughly with deionized or distilled water. If sampling from a tap, allow the water to flow at least 5minutes to ensure a representative sample.  Allow several volumes of water to overflow the container and cap the container so there is no headspace					

Chlorine, Total

# 1

# Procedures DPD Method



 Fill a sample cell with 10 mL of sample. (the prepared sample)



Add the contents of one DPD Total
 Chlorine Reagent Pillow to the sample cell.
 Swirl the sample cell for 20 seconds
 to mix. A 3 minute reaction period will begin. Perform next steps during this period.



3. Fill a second sample cell with 10 mL of sample.(the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 9.** 

(HACH DR/890 : 9

DR/2010 & 2500 : 80

DR/4000 : 1450)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



6. Within 3 minutes after the timer beep, wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Cl<sub>2</sub>)

Chlorine, Total  $oldsymbol{8}$ 



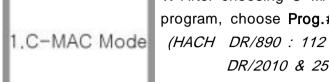
# Chlorine Dioxide (0.04 $\sim$ 5.00 mg/L ClO $_2$ )

# DPD Method

Required Reagents	DPD Free Chlorine Re Glycine Reagent	agent Pillow	Cat. NO.	11430-00
	Acidity	Greater than 150 mg/L CaCO <sub>3</sub>	Cr <sup>6+</sup>	Greater than 2 mg/L
	Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub>		Copper: Greater than 10 mg/L
	Bromine	At all levels	Metals (Cl <sub>2</sub> : 0.6mg/L)	Nickel: Greater than 50 mg/L Other metals may also interfere
	Chlorine	Greater than 6 mg/L	(012. 0.0111972)	: add more glycine
	Chloramines, organic	At all levels		When read within 1 minutes after reagent
Interferences	Flocculating agents	Greater than Al(SO <sub>4</sub> ) <sub>3</sub> 500 mg/L		addition, 3mg/L monochloramine causes
	(Cl <sub>2</sub> : 0.6mg/L) Greater than FeCl <sub>2</sub> 200 mg/L		less than a 0.1 mg/L increase in the reading	
	Hardness	Greater than 1000 mg/L as CaCO <sub>3</sub>	Ozone	Greater than 1.5mg/L
	lodine	At all levels	Peroxides	At all levels
	Mn <sup>4+</sup> Mn <sup>7+</sup> At all levels		Extreme sample pH or highly buffered samples	Neutralize to pH 6~7
Sampling Storage & Preservation	(Bleach 1mL per liter) water to flow at least	for at least 1hour. Rinse thorough	ly with deionized or distiller re sample. Allow several v	demand by soaking in a dilute bleach solutioned water. If sampling from a tap, allow the olumes of water to overflow the container bles immediately. Do not preserve.
Tips & Techniques	dioxide in water. For using deionized water	more accurate results, determine a in place of the sample. Subtract t	reagent blank value for e the reagent blank value fro	linity influence decomposition of chlorine ach new lot of reagent. Follow the procedure om the final results of perform a reagent blanke color may fade or the sample turn yellow.

Chlorine Dioxide

#### **DPD** Method



1. After choosing C-MAC mode in the program, choose Prog.# 101.

DR/2010 & 2500 : 76

DR/4000 : 1530)



2. Fill a sample cell with 10 mL of sample. (the prepared sample)

Fill a second sample cell with 10 mL of sample. (the blank)



3. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



4. Add 4 drops of Glycine Reagent to the sample cell. Swirl to mix.



5. Add the contents of one DPD Free Chlorine Reagent Pillow to the sample cell.(the prepared sample) Cap the cell and swirl to mix. Wait 30 seconds for undissolved powder to settle.



6. Within 1 minute of adding the reagent, wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter. (Results will appear in mg/L ClO<sub>2</sub>)

10 Chlorine Dioxide



Chromium Hexavalent (0.01~0.60 mg/L Cr<sup>6+</sup>)

1,5-Diphenylcarbohydrazide Method

Required Reagents	Chromium 3	Reagent Pillow	Cat. NO.	11510-00		
	Iron	May interfere above 1 m	g/L			
	Mercurous & Mercuric Ions	slightly				
Interferences	рН	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.				
	Vanadium	May interfere above 1 mg/L				
	Turbidity	For turbid samples, treat the blank with the contents of one Acid Reagent Powder Pillow. This will ensure that any turbidity dissolved by the acid in the Chromium 3 Reagent. Chromium 3 Reagent will also be dissolved in the blank.				
Sampling,Storage & Preservation		es in a cleaned glass or posterior be analyzed within 24 ho		Store at 4 °C (39 °F) up to 24 hours.		
Tips & Techniques	At high chromium levels, a precipitate will form.  The final samples are highly acidic. Neutralize to pH 6.9 with NaOH Standard Solution and flush down the drain for disposal. For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.					

Chromium, Hexavalent 11

## 1,5-Diphenylcarbohydrazide Method



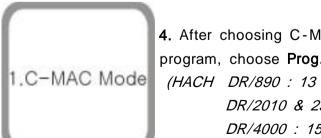
1. Fill a sample cell with 10 mL of sample. (the prepared sample)



2. Add the contents of one Chromium 3 Reagent Pillow to the sample cell. Cap and invert gently to mix. A 5 minute reaction period will begin. (A purple color will form if hexavlent chromium is present.)



3. Fill a second sample cell with 10 mL of sample. (the blank)



4. After choosing C-MAC mode in the program, choose Prog.# 13.

DR/2010 & 2500 : 90

DR/4000 : 1560)



5. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



6. Wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Cr<sup>6+</sup>)

12 Chromium, Hexavalent



Chromium, Total (0.01  $\sim$  0.60 mg/L Cr)

# Alkaline Hypobromite Oxidation Method

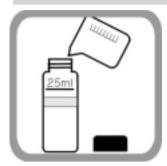
Required Reagents	Acid Reagent Pillow Chromium 3 Reagent Pillow Chromium 1 Reagent Pillow Chromium 2 Reagent Pillow		Cat. NO.	11520-00			
			may exceed the buffering capacity of the reagents and require sample pretreatment.				
Interferences	Organic material  May inhibit complete oxidation of trivalent chromium.  If high levels of organic material are present, digestion is require						
	Turbidity		For turbid samples, treat the 25-mL blank and the sample the same during steps 1-6				
Sampling Storage & Preservation	Collect samples in acid-washed glass or plastic containers. To preserve samples adjust the pH to 2 or less with nitric acid (about 2mL per liter). Store preserved samples at room temperature up to six months. Adjust the pH to about 4 with 5.0 N NaOH.						
Tips & Techniques	Undissolved powder does not affect accuracy.  Prepare a boiling water bath. For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.  Use finger cots to handle hot sample cells						

Chromium, Total

# ¥

#### **Procedures**

## **Alkaline Hypobromite Oxidation Method**



1. Fill a sample cell with 25 mL of sample.



Add the contents of one Chromium 1
Reagent Pillow to the sample cell.
 Cap and invert gently to mix.
 (the prepared sample)



- **3.** Remove the cap and place the prepared sample into a boiling water bath.
- A 5 minute reaction period will begin.



**4.** When the timer beeps, remove the prepared sample. Using running water, cool the cell to **25**. Be sure the caps are on tightly.



5. Add the contents of one Chromium 2Reagent Pillow to the sample cell.Cap and invert gently to mix.



6. Add the contents of one Acid Reagent Pillow to the sample cell.Cap and invert gently to mix.



- **7.** Add the contents of one Chromium **3** Reagent Pillow to the sample cell.
- A 5 minute reaction period will begin.

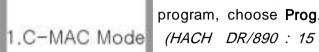


**8.** Fill a second sample cell with **25 mL** of sample. (the blank)

Chromium, Total

# **Alkaline Hypobromite Oxidation Method**

**Procedures** 



9. After choosing C-MAC mode in the program, choose Prog.# 15.

DR/2010 & 2500 : 100

DR/4000 : 1580)



10. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



11. Wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Cr)

*15* Chromium, Total



# Copper (0.04 $\sim$ 5.00 mg/L Cu)

# Bicinchoninate Method

Required Reagents	Copper Reager	nt Pillow	Cat. NO.	11710-00		
	Acidity	•	``	ess) a precipitate may form. Add swirling to dissolve the turbidity		
	Aluminum, Al <sup>3+</sup>	powder A for Copper R	eagent Pillow. Result	but substitute a copper reagent s obtained will include total quires a 25 mL sample cell.		
Interferences	Cyanide, CN <sup>-</sup>	to the 10 mL sample.	Before adding the Copper reagent powder, add 0.2 mL of formaldehyde to the 10 mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde			
	Hardness	Substitute a copper reagent powder A for Copper Reagent Pillow				
	Iron, Fe <sup>3+</sup>	Substitute a copper rea	gent powder A for C	Copper Reagent Pillow		
	Silver, Ag <sup>+</sup>	10 drops of saturated h	KCI solution to 75 m	interference is likely. Add L of sample, followed by filtering the filtered sample in the		
Sampling Storage & Preservation	the pH to 2 or Store preserved	Collect samples in acid-washed glass or plastic containers. To preserve samples adjust the pH to 2 or less with nitric acid (about 2mL per liter).  Store preserved samples at room temperature up to six months. Before analysis, adjust the pH to 4.6 with 8 N KOH. Do not exceed pH 6, as copper may precipitate.				
Tips & Techniques	Digestion is required for determining total copper.  Accuracy is not affected by undissolved powder.					

# 1

#### Procedures

#### Bicinchoninate Method



1. Fill a sample cell with 10 mL of sample.



 Add the contents of one Copper Reagent Pillow to the sample cell.
 Cap and invert gently to mix.
 (the prepared sample)

A 2 minute reaction period will begin.



**3.** Fill a second sample cell with **10 mL** of sample. (the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 20**.

(HACH DR/890 : 20

DR/2010 & 2500 : 135

DR/4000 : 1700)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**6.** Within **30 minutes** after the timer beeps, wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

Press Enter.

(Results will appear in mg/L Cu)



Copper (0.002~0.210 mg/L Cu)

# Porphyrin Method

Required Reagents	Copper Masking Reagent Pillow Porphyrin 1 Reagent Pillow Porphyrin 2 Reagent Pillow		Cat. NO.	11720-00	
	Aluminum, Al <sup>3+</sup>	60 mg/L	Manganese	140 mg/L	
	Cadmium, Cd	10 mg/L	Mercury, Hg <sup>2+</sup>	3 mg/L	
	Calcium, Ca <sup>2+</sup>	1500 mg/L	Molybdenum	11 mg/L	
	Chloride, Cl⁻	90,000 mg/L	Nickel, Ni <sup>2+</sup>	60 mg/L	
	Chromium, Cr <sup>6+</sup>	110 mg/L	Potassium, K <sup>+</sup>	60,000 mg/L	
Interferences	Cobalt, Co <sup>2+</sup>	100 mg/L	Sodium, Na <sup>+</sup>	90,000 mg/L	
	Fluoride, F	30,000 mg/L	Zinc, Zn <sup>2+</sup>	9 mg/L	
	Iron, Fe <sup>2+</sup>	6 mg/L	Chelating agents	Unless digestion is performed	
	Lead, Pb <sup>3+</sup>	3 mg/L	Extreme sample pH or	may exceed the buffering capacity of the	
	Magnasium	10,000 mg/L	highly buffered samples	reagents and require sample pretreatment	
Sampling Storage & Preservation	Collect samples in acid-washed glass or plastic containers. To preserve samples adjust the pH to 2 or less with nitric acid. (about 5mL per liter). Store preserved samples at room temperature up to six months at room temperature. Before testing, adjust the pH of the preserved sample to between 2-6. If the sample is too acidic, adjust the pH with 5.0 N NaOH solution				
Tips & Techniques	water. Rinse aga	Digestion is required for determining total copper Wash all glassware with detergent. Rinse with tap water. Rinse again with 1:1 Nitric Acid Solution. Rinse a third time with 1:1 Nitric Acid Solution. Rinse a third time with copper-free, deionized water.			

### Porphyrin Method



1. Fill two round sample cells with 10 mL of sample.



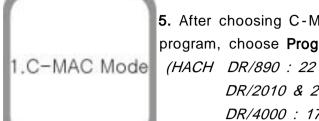
2. Add the contents of one Copper Masking Reagent Pillow to the sample cell. Cap and invert gently to mix. (the blank) The second sample cell is the prepared sample.



3. Add the contents of one Porphyrin 1 Reagent Pillow to each sample cell. Cap and invert gently to mix.



4. Add the contents of one Porphyrin 2 Reagent Pillow to each sample cell. Cap and invert gently to mix. If copper is present, the sample will turn blue mometarily, then return to yellow. A 3 minute reaction period will begin.



5. After choosing C-MAC mode in the program, choose Prog.# 22.

DR/2010 & 2500 : 145

DR/4000 : 1720)



6. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



7. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Cu)



# Cyanide (0.001 $\sim$ 0.240 mg/L CN $^{-}$ )

# **Pyridine-Pyrazalone Method**

Required Reagents	Cyanide 2	1 Reagent Pillow 2 Reagent Pillow	Cat. NO.	11810-00			
	Cyanide 3	3 Reagent Pillow					
	Chlorine	Large amounts of chlorine in the sample will cause a milky white precipitate after the addition of the Cyanide 3 reagent. If chlorine or other oxidizing agents are known to be present, pretreat the sample before testing using the procedure in this table for oxidizing agents.					
	Metals	Ni or Co up to 1 mg/L do not interfere. Eliminate the interference from up to 20 mg/L Cu and 5 mg/L iron by adding the contents of one Chelating Reagent Powder Pillow to the sample and then mixing before adding Cyanide 1 Reagent Powder Pillow. Prepare a reagent blank of deionized water and reagents to zero the instrument.					
Interferences	Oxidizing Agents	Adjust a 25 mL portion of the alkaline sample to pH 7-9 with 2.5 N HCl Standard Solution.  Count the number of drops of acid added.  Add 2 drops of Kl Solution and 2 drops of Starch Indicator Solution to the sample.  Swirl to mix. The sample will turn blue if oxidizing agents are present.  Add Sodium Arsenite Solution drop-wise until the sample turns colorless.  Swirl the sample thoroughly after each drop. Count the number of drops.  Take another 25 mL sample and add the total number of drops of HCl Standard Solution counted in step.  Subtract one drop from the amount of Sodium Arsenite Solution added in step. Add this amount to the sample and mix thoroughly. Continue with step. of the cyanide procedure.					
	Reducing Agents	Adjust a 25 mL portion of the alkaline sample to pH 7-9 with 2.5 N HCl Standard Solution.  Count the number of drops of acid added.  Add 4 drops of Kl Solution and 4 drops of Starch Indicator Solution to the sample.  Swirl to mix. The sample should be colorless.  Add Bromine Water drop-wise until a blue color appears. Swirl the sample thoroughly after each addition. Count the number of drops.  Take another 25 mL sample and add the total number of drops of HCl Acid Standard Solution counted in step Add the total number of drops of Bromine Water counted in step c to the sample and mix thoroughly. Continue with step of the cyanide procedure.					
	Turbidity	Large amounts of turbidity will cause high returbid water samples. The test results should	•				

Cyanide 21

# Tips & Techniques

Use a water bath to maintain the optimum temperature for the reaction in this test (25 °C). Samples at less than 23 °C require longer reaction times, and samples at greater than 25 °C yield low results. longer reaction times, and samples at greater than 25 °C yield low results water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. The timing of reagent adding is critical. You may find it useful to open the necessary reagents before starting this sequence. All samples to be analyzed for cyanide should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation.

#### Sampling, Storage

&

#### Preservation

Collect samples in glass or plastic bottles and analyze as quickly as possible. The presence of oxidizing agents, sulfides and fatty acids can cause the loss of cyanide during sample storage. Samples containing these substances must be pretreated as described below before preservation with NaOH. If the sample contains sulfide and is not pretreated, it must be analyzed within 24 hours. Preserve the sample by adding 4.0 mL of 5.0 N NaOH to each liter(or quart) of sample, using a glass serological pipet and pipet filler. Store the samples at 4 °C (39 °F) or less. Samples preserved in this manner can be stored for 14days. Before testing samples should be adjusted to approximately pH 7 with 2.5 N HCI.

With most compounds, a one-hour reflux is adequate. If thiocyanate is present in the original sample, a distillation step is absolutely necessary as thiocyanate causes a positive interference. High concentrations of Sulfidethiocyanate can yield a substantial quantity of sulfide in the distillate. The "rotten egg" smell of hydrogen sulfide will accompany the distillate when sulfide is present. The sulfide must be removed from the distillate prior to testing. If cyanide is not present, the amount of thiocyanate can be determined. The sample is not distilled and the final reading is multiplied by 2.2. The result is mg/L SCN. The distillate can be tested and treated for sulfide after the last step of the distillation procedure by using the following lead acetate treatment procedure.

# Acid Distillation

Place a drop of the distillate (already diluted to 250 mL) on a disc of Hydrogen Sulfide Test Paper that has been wetted with pH 4.0 Buffer Solution.

If the test paper darkens, add 2.5 N HCl Standard Solution. drop-wise to the distillate until a neutral pH is obtained.

Add a 1-g measuring spoon of lead acetate to the distillate and mix. Repeat step 1.

If the test paper continues to turn dark, keep adding lead acetate until the distillate tests negative for sulfide. Filter the black lead sulfide precipitate through filter paper and a funnel. Neutralize the liquid filtrate to pH 7 and immediately analyze for cyanide.

#### **Acid Distillation Procedure**

Fill a 100 mL sample(below 0.05 mg CN) at 500 mL distillation flask and dilute to 250 mL with deionized water.

Adding 2~3 drops phenolphthalein ethylalcohol solution(0.5 W/V%) as indicator.

Neutralize with phosphoric acid or 2% NaOH solution and set up cyanide distillatilling apparatus.

Adding ammonium sulfamate solution(10 W/V%) 1mL ,phosphoric acid 10 mL and EDTA solution(for cyanide test)

10 mL. Waiting for several minute. Heat the flask (Distillating velocity: 2~3 mL/min)

Collecting the distillate at 100 mL mass cylinder filled with 2% NaOH 20mL until volume is 90 mL

Seperate the condenser and rinse the inside of condeser with deionized water. Dillute to 100 mL.

Cyanide 22

## **Pyridine-Pyrazalone Method**



1. Using a graduated cylinder, fill a round sample cell with a 10 mL of sample. (the prepared sample)



2. Add the contents Cyanide 1 Reagent Pillow. Cap and shake for 30 seconds. Leave the sample cell undisturbed for an additional 30 seconds.



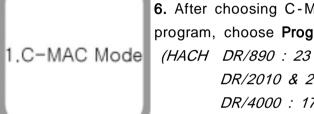
3. Add the contents Cyanide 2 Reagent Pillow. Cap and shake for 10 seconds. Immediately proceed to next step. (Delaying the addition of the Cyanide 2 reagent will produce low test results.)



4. Add the contents Cyanide 3 Reagent Pillow. Cap and shake vigorously. A 30 minute reation period will begin. (If cyanide is present, the solution will turn from pink to blue)



5. Fill another round sample cell with **10 mL** of sample.(The blank)



6. After choosing C-MAC mode in the program, choose Prog.# 23.

DR/2010 & 2500 : 160

DR/4000 : 1750)



7. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



8. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L CN<sup>-</sup>)

23 Cyanide



# Cyanuric Acid (5~50 mg/L Cyan Acid)

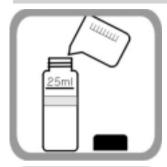
# **Turbidimetric Method**

Required Reagents	Cyanuric Acid Reagent Pillow	Cat. NO.	11910-00		
Sampling, Storage & Preservation	Collect samples in clean plastic or glass bottles. Samples must be analyzed within 24 hours.				
Tips & Techniques	Filter highly turbid samples with filter paper and a funnel.  After adding the reagent, a white turbidity will form if cyanuric acid is present.  Clean sample cells with soap, water, and a brush soon after each test to avoid a build-up of film on the sample cell.				

# 1

#### Procedures

#### **Turbidimetric Method**



1. Fill a sample cell with 25 mL of sample.



2. Add the contents Cyanuric Acid Reagent Pillow. Cap and swirl to mix.

(the prepared sample).

A 3 minute reation period will begin.



3. Fill another round sample cell with25 mL of sample.(the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 24.** 

(HACH DR/890 : 24

DR/2010 & 2500 : 170)



5. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**6.** Within **7 minutes** after the timer beeps, wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Cyan Acid)

Cyanuric Acid 26



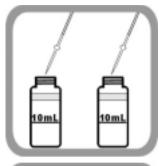
# Fluoride ( $0.02\sim2.00$ mg/L F )

# SPADNS Method

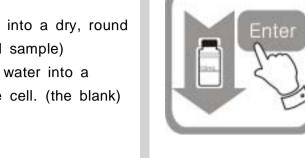
Required Reagents	SPADNS reagent solution		Cat. NO.	13310-00		
	Alkalinity (as CaCO <sub>3</sub> )	At 5000 mg/L it causes a -0.1 mg/L F error				
	Aluminum	At 0.1 mg/L it causes a .0.1 mg/L F error. To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting 2 hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.				
	Chloride	At 7000 mg/L it causes a +0.1 mg/L F <sup>-</sup> error				
Interferences	Chlorine	SPADNS Reagent contains enough arsenite to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, add one drop of Sodium Arsenite Solution to 25 mL of sample for each 2 mg/L of Chlorine.				
	Iron, Ferric	At 10 mg/L it causes a -0.1 mg/L F <sup>-</sup> error				
	Phosphate, ortho	At 16 mg/L it causes a +0.1 mg/L F <sup>-</sup> error				
	Sodium Hexameta - phosphate	At 1.0 mg/L it causes a +0.1 mg/L F <sup>-</sup> error				
	Sulfate	At 200 mg/L it causes a +0.1 mg/L F <sup>-</sup> error				
Sampling, Storage & Preservation	Samples may be stored in glass or plastic bottles for at least seven days when cooled to 4 °C (39 °F) or lower. Warm samples to room temperature before analysis.					
Tips & Techniques	Distillation is required. Interference is eliminated mostly in this procedure.  The sample and deionized water should be at the same temperature (±1 °C). Temperature adjustments may be made before or after reagent addition.  Fluoride reagent solution is toxic and corrosive.					

Fluoride 27

SPADNS Method



1. Pipet 10 mL of sample into a dry, round sample cell. (the prepared sample) Pipet 10 mL of deionized water into a second dry, round sample cell. (the blank)



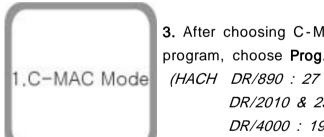
5. Wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L F<sup>-</sup>)



2. Carefully pipet 2 mL of SPADNS Solution into each cell. Swirl to mix.

A 1 minute reaction period will begin.



3. After choosing C-MAC mode in the program, choose Prog.# 27.

DR/2010 & 2500 : 190

DR/4000 : 1900)



4. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.

28 Fluoride



Hardness (0.07 $\sim$ 4.00 mg/L Ca and Mg as CaCO<sub>3</sub>)

**Calmagite Colorimetric Method** 

Required Reagents	Alkali Solution for Ca and Mg Indic EDTA Solution, C EGTA Solution		Cat. NO.	12010-00		
	Cr <sup>3+</sup>	Above 0.25 mg/L				
	Cu <sup>2+</sup>	Above 0.75 mg/L				
	EDTA, chelated	Above 0.2 mg/L (as CaCO <sub>3</sub> )				
	EDTA or EGTA	Traces remaining in sample cells from previous tests will give erroneous results.  Rinse cells thoroughly before using.				
Interferences	Fe <sup>2+</sup>	Above 1.4 mg/L				
IIIlerrererices	Fe <sup>3+</sup>	Above 2.0 mg/L				
	Mn <sup>2+</sup>	Above 0.2 mg/L				
	Zn <sup>2+</sup>	Above 0.05 mg/L				
	Ca>1.0 mg/L Mg>0.25 mg/L	if the calcium is over 1.0 and the magnesium is over 0.25 mg/L as CaCO <sub>3</sub> .				
Sampling Storage & Preservation	Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with Nitric Acid (about 5 mL per liter). Cool samples to 4 °C.Preserved samples can be stored up to six months. Before analysis, adjust the sample pH to between 3 and 8 with 5.0 N NaOH Solution.					
Tips & Techniques	For the most accurate magnesium test results, keep the sample temperature between 21 ~ 29 . The test will detect any calcium or magnesium contamination in the mixing cylinder, measuring droppers, or sample cells. To test cleanliness, repeat the test until result are consistent. Total hardness in mg/L equals mg/L Ca as CaCO <sub>3</sub> plus mg/L Mg as CaCO <sub>3</sub> . Remaining traces of EDTA or EGTA from previous tests will give erroneous results. Rinse sample cells thoroughly before using.					

Hardness 29

## **Calmagite Colorimetric Method**



1. Pour 100 mL of sample into a 100 mL graduated mixing cylinder.



2. Add 1 mL of Calcium and Magnesium Indicator Solution. Stopper the cylinder and invert it several times.



3. Add 1 mL of Alkali Solution for Calcium and Magnesium Test. Stopper the cylinder and invert it several times.



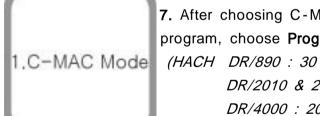
4. Pour 25 mL of the solution into each of three, round sample cells.



5. Add one drop of EDTA Solution to the first cell(the blank). Swirl to mix.



6. Add one drop of EGTA Solution to the second cell. Swirl to mix.



7. After choosing C-MAC mode in the program, choose Prog.# 30.

DR/2010 & 2500 : 225

DR/4000 : 2020)



8. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.

30 Hardness

### **Calmagite Colorimetric Method**

Procedures



**9.** Wipe the second cell and place it into the cell holder. Place the cover on the sample cell. Press Enter. (Results will appear in mg/L Mg as CaCO<sub>3</sub>)



Do not remove the cell from the instrument. Record the results. Press ESC.
 In program, choose Prog.# 29.

(HACH DR/890 : 29

DR/2010 & 2500 : 220

DR/4000 : 2010)



11. Press Zero.



**12.** Wipe the third cell and place it into the cell holder. Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Mg as CaCO<sub>3</sub>)

Total hardness

= Ca as CaCO<sub>3</sub> + Mg as CaCO<sub>3</sub>

Hardness 31

# mac

# Iron $(0.009 \sim 1.300 \text{ mg/L Fe})$

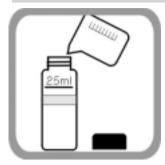
# iron Method

Required Reagents	Iron Reagent Solution		Cat. NO.	12210-00		
	Strong chelants (EDTA)	At all levels				
	Cobalt	May give slightly high results				
	Copper	May give slightly high results				
Interferences	Hydroxides, Rust	After Adding Iron Reagent, Heat in a boiling water bath for 1 minute.  Cool to 24 before proceeding next step. Return the sample volume to 25mL deionized water.				
	Pour 25mL of sample in 125mL flask. Add the contents of Iron Reagent Solution and swirl to mix. Boil gently for 20~30 minutes.  Do not allow to boil dry. A purple color will develop if iron is present.  Return the boiled sample to the 25-mL graduated cylinder Return the sample volume to the 25-mL mark with deionized water. Pour this solution into a sample cell and swirl to mix. Proceed with step3					
Sampling Storage & Preservation	Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with concentrated Nitric Acid(about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If you are only reporting dissolved iron, filter the sample immediately after collection and before adding nitric acid. Before testing, adjust the sample pH to 3.5 with Ammonium Hydroxide. Do not exceed pH 5, or iron may precipitate					
Tips & Techniques	Digestion is required for total iron determination. Rinse glassware with a 1:1 HCl and rinse again with deionized water. For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.					

Iron 33

## Ŧ

## Procedures iron Method



1. Fill a sample cell with 25 mL of sample. (the prepared sample)



2. Add the contents of one Iron Reagent Solution Pillow to the sample cell.

Cap and mix.

A **5 minute** reacition period will begin.

(A violet color will develop if iron is present)



3. Fill another sample cell with 25 mL of sample. (the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 37**.

(HACH DR/890 : 37

DR/2010 & 2500 : 260

DR/4000 : 2175)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**6.** Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

Press Enter.

(Results will appear in mg/L Fe)

Iron 34



Iron, Ferrous (0.02 $\sim$ 3.00 mg/L Fe<sup>2+</sup>)

1,10 Phenanthroline Method

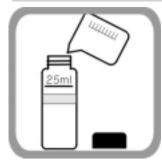
Required Reagents	Ferrous Iron Reagent Pillow	Cat. NO.	12310-00		
Sampling, Storage & Preservation	Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection.				
Tips & Techniques	For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.				

Iron, Ferrous 35

## Ŧ

#### **Procedures**

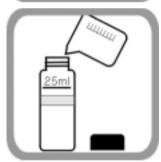
#### 1,10 Phenanthroline Method



1. Fill a sample cell with 25 mL of sample. (the prepared sample)



2. Add the contents of one Ferrous Iron Reagent Pillow to the sample cell. Cap and mix. A 3 minute reacition period will begin. (An orange color will develop if Ferrous Iron is present)



3. Fill another sample cell with 25 mL of sample. (the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 33**.

(HACH DR/890 : 33

DR/2010 & 2500 : 255

DR/4000 : 2150)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**6.** Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Fe<sup>2+</sup>)

iron, Ferrous 36



## Iron, Total (0.02 $\sim$ 3.00 mg/L Fe)

## Total Iron Method

Required Reagents	Total Iron Reagent Pillov	N	Cat. NO.	12410-00		
	Ca <sup>2+</sup>	No effect at less than 10,000 mg/L as CaCO <sub>3</sub>				
	CI <sup>-</sup>	No effect at less	than 185,000 mg/L			
	High Iron Levels	Inhibit color deve	lopment. Dilute sam	ple and re-test to verify results.		
	Iron Oxides	After mild, vigoro	us digestion, adjust	sample to pH 3 ~ 5 with NaOH Solution.		
	Magnesium	No effect at 10,0	000 mg/L as CaCO <sub>3</sub>			
	Molybdate Molybdenum	No effect at 50 r	ng/L as Mo			
Interferences	High Sulfide Levels	250mL Erlenmeye	er flask. Boil 20 minu	I area. Add 5 mL HCl to 100mL sample in a utes and cool. Adjust pH to 3 ~ 5 with NaOH with deionized water.		
	Turbidity	Add 0.1 g scoop of Rust Remover to the blank. Swirl to mix. Zero the instrument with this blank. If sample remains turbid, add three 0.2 g scoops of Rust Remover to a 75-mL sample. Let stand 5 minutes. Filter through a glass membrane filter.				
	Extreme sample pH or highly buffered samples	Adjust pH to 3 ~ 5.				
Sampling Storage & Preservation	Collect samples in acid-washed glass or plastic bottles. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the sample pH to 2 or less with concentrated Nitric Acid (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. Before testing, adjust the sample pH to 3 ~ 5 with Ammonium Hydroxide.					
Tips & Techniques	Digestion is required for total iron determination. Accuracy is not affected by undissolved powder. For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.					

iron, Total 37

## 1

## Procedures Total Iron Method



1. Fill a sample cell with 10 mL of sample. (the prepared sample)



2. Add the contents of one Total Iron Reagent Pillow to the sample cell. Cap and mix. A 3 minute reacition period will begin. Allow samples that contain rust to react for at least 5 minutes.



3. Fill another sample cell with 10 mL of sample. (the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 33**.

(HACH DR/890 : 33

DR/2010 & 2500 : 265

DR/4000 : 2165)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**6.** Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Fe)

Iron, Total



## Manganese, LR (0.007~0.700 mg/L Mn)

PAN Method

Required Reagents	Cyanide Reagent Ascorbic Acid Powder Pillow PAN Indicator Solution, 0.1%		Cat. NO.	12511-00	
	Aluminum	20 mg/L	Lead	0.5 mg/L	
	Cadmium	10 mg/L	Magnesium	300 mg/L as CaCO₃	
	Calcium	1000 mg/L as CaCO <sub>3</sub>	Nickel	40 mg/L	
Interferences	Cobalt	20 mg/L	Zinc	15 mg/L	
	Copper	50 mg/L			
	Iron 25 mg/L (If sample contains more than 5 mg/L iron, allow a 10-minute reaction period in step 5.)				
Sampling Storage & Preservation	to 2 or less wit	Collect samples in clean plastic bottles. To preserve samples, adjust the sample pH to 2 or less with concentrated Nitric Acid(about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. Before testing, adjust the sample pH to 4 ~ 5 with 5N NaOH.			
Tips & Techniques	Digestion is required for determining total manganese.  Rinse all glassware with 1:1 Nitric Acid Solution. Rinse again with deionized water.  The alkaline cyanide solution contains cyanide. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas.				

Manganese, LR 39

#### PAN Method



1. Fill a sample cell with 10 mL of deionized water.(the blank)



2. Fill another sample cell with 10 mL of sample. (the prepared sample)



3. Add the contents of one Ascorbic Acid Powder Pillow to each cell. Cap and mix gently.



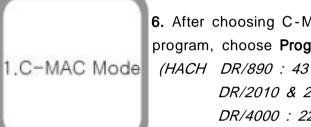
**4.** Add **15 drops** of (0.6 mL) akaline - cyanide Reagent Solution to each cell. Cap and mix gently. A cloudy solution may form.

The turbidity should dissipate after step 5.



**5.** Add **21 drops** of (0.8 mL) 0.1% PAN Indicator solution to each cell. Cap and mix gently. An orange color will develop if manganese is present.

A 2 minute reaction period will begin.



6. After choosing C-MAC mode in the program, choose Prog.# 43.

DR/2010 & 2500 : 290

DR/4000 : 2260)



7. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



8. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Mn)

40 Manganese, LR



## Manganese, HR (0.2~20.0 mg/L Mn)

## Periodate Oxidation Method

Required Reagents	Buffer Pillow Sodium Periodate Pillow		Cat. NO.	12510-00		
	Calcium	700 mg/L				
	Chloride	70,000 mg/L				
Interferences	Iron	5 mg/L				
	Magnesium	100,000 mg/L				
	рН		Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment			
Sampling Storage & Preservation	Collect samples in acid-washed glass or plastic bottles.( Do not glass bottle) If samples are acidified, adjust the pH 4 ~ 5 with 5N NaOH before analysis. Do not exceed pH 5, as manganese may precipitate.					
Tips & Techniques	Digestion is required for determining total manganese.  For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.					

Manganese, HR 41

#### Periodate Oxidation Method



1. Fill a sample cell with 10 mL of sample. (the prepared sample)



2. Add the contents of one Buffer Pillow to each cell. Cap and mix gently.

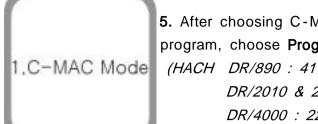


3. Add the contents of one Sodium Periodate Pillow to each cell. Cap and mix gently. A violet color will develop if manganese is present.

A 2 minute reaction period will begin.



4. Fill another sample cell with 10 mL of sample. (the blank)



5. After choosing C-MAC mode in the program, choose Prog.# 41.

DR/2010 & 2500 : 295

DR/4000 : 2250)



6. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



7. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Mn)

42 Manganese, HR



## Nitrate, HR (0.2 $\sim$ 30.0 mg/L NO<sub>3</sub>-N)

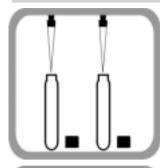
## Chromotropic Acid Method

Required Reagents	Nitrate, HR Vial Nitrate Reagent 1 Pillow (Chromotropic Acid Method)		Cat. NO.	10413-00	
	Barium	A negative interference at concentrations greater than1 mg/L			
	Chloride	Does not inte	erfere below 1000	mg/L	
Interferences	Nitrite	A positive interference at concentrations greater than 12 mg/L. Remove nitrite interference up to 100 mg/L by adding 400mg of urea to 10mL of sample. swirl to dissolve.			
	Copper	Positive at all levels.			
Sampling Storage & Preservation	Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with Concentrated Sulfuric Acid(about 2 mL per liter). Sample refrigeration is still required. Before testing the stored sample, warm to room temperature and neutralize with 5N NaOH solution.  Do not use mercury compounds as preservatives.				
Tips & Techniques	This test is technique-sensitive. Invert the vials as described here to avoid low results: Hold the vial in a vertical position with the cap pointing up. Turn the vial upside-down. Wait for all of the solution to flow down to the cap. Pause. Return the vial to an upright position. Wait for all the solution to flow to the bottom of the vial. This process equals one inversion.				

### Property

#### **Procedures**

## Chromotropic Acid Method



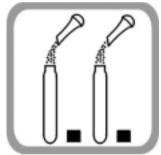
 Add 1 mL of sample to one Nitrate, HR (Chromotropic Acid Method) Vial.

(the prepared sample)

Add **1 mL** of deionized water to another vial. (the blank)



2. Cap vials and invert 10 times to mix.



**3.** Add the contents of Nitrate(Chromotropic Acid Method) Reagent Pillow to each vial.



**4.** Cap vials and invert 10 times to mix. Some solid matter will not dissolve.

A 5 minute reaction period will begin.

Do not invert the vial again.

A yellow color will develop if Nitrate Nitrogen is present.



**5.** After choosing C-MAC mode in the program, choose **Prog.# 57.** 

(HACH DR/890 : 57

DR/2010 & 2500 : 344

DR/4000 : 2511)



**6.** Wipe the blank and place it into the cell holder. Place the cover on the vial. Press Zero.



7. Within 5 minutes wipe the prepared sample and place it into the cell holder. Place the cover on the vial.

Press Enter.

(Results will appear in mg/L NO<sub>3</sub>-N)

Nitrate, HR 44



## Nitrate, LR (0.01 $\sim$ 0.50 mg/L NO<sub>3</sub>-N)

## Cadmium Reduction Method

Required Reagents	Nitrate LR Rea	gent Pillow (Cadmium Reduction Method) gent Pillow	Cat. NO.	10422-11		
	Calcium	100 mg/L				
	Chloride	Concentrations Above 100 mg/L will cause a calibration must be done using standar		•		
	Ferric iron	At all levels				
Interferences	Nitrite		the sample. Pre of nitrite found for the sample in st	ep 2 until a yellow color remains.		
	рН	PH Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment				
Sampling Storage & Preservation	More reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 48 hours at 4 °C. To preserve samples for longer periods, add 2mL of sulfuric acid per liter and store at 4°C. Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5N NaOH solution. Do not use mercury compounds as preservatives.					
Tips & Techniques	For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust. Rinse the sample cell and mixing cylinder immediately after use to remove all cadmium particles.  A deposit of unoxidized metal will remain after the Nitrate LR Reagent Pillow dissolves. The deposit will not affect results. Shaking time and technique influence color development. Analyze a standard solution several times and adjust the shaking time to obtain the correct result. Use this time for analyzing samples					

#### Cadmium Reduction Method



1. Fill a 25 ml graduated mixing cylinder with 15 mL of sample.



2. Add the contents of one Nitrate LR Reagent Pillow to the cylinder. Stopper. Shake the cylinder vigorously for 3 minutes. A 2 minute reaction period will begin.



3. When the timer beeps, carefully pour 10 mL of the sample into sample cell. Do not transfer any cadmium particles to the sample cell.

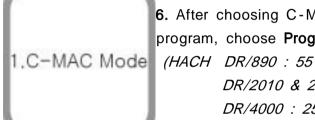


4. Add the contents of Nitrite LR Reagent Pillow to each cell. Cap and mix gently. (the prepared sample)

A pink color develop if nitrate is present. A 15 minute reaction period will begin.



5. Fill a second sample cell with 10 mL of original sample.(the blank)



6. After choosing C-MAC mode in the program, choose Prog.# 55.

DR/2010 & 2500 : 351

DR/4000 : 2515)



7. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



8. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L NO<sub>3</sub>-N)



## Nitrate, MR (0.1 $\sim$ 5.0 mg/L NO<sub>3</sub>-N)

## Cadmium Reduction Method

Required Reagents	Nitrate MR Re	agent Pillow (Cadmium Reduction Method)	Cat. NO.	10423-11		
	Calcium	100 mg/L				
	Chloride	Concentrations Above 100 mg/L will caus a calibration must be done using standard				
	Ferric iron	At all levels				
Interferences	At all levels: This method measures both the nitrate and nitrite in the sample. If nitrite is prese LR Test (Prog.# 60) should be done on the sample. Pretreat the nitrate nitrogen sample with the pretreatment. Then subtract the amount of nitrite found from the results of the NO <sub>2</sub> -N, LR Test Add 30-g/L Bromine Water dropwise to the sample in step 2 until a yellow color remains. Mix after each drop. Add one drop of 30-g/L Phenol Solution to destroy the color.					
	рН	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment				
Sampling Storage & Preservation	More reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 48 hours at 4 °C. To preserve samples for longer periods, add 2mL of sulfuric acid per liter and store at 4°C. Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5N NaOH solution. Do not use mercury compounds as preservatives.					
Tips & Techniques	For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust. Rinse the sample cell and mixing cylinder immediately after use to remove all cadmium particles.  A deposit of unoxidized metal will remain after the Nitrate MR Reagent Pillow dissolves. The deposit will not affect results. Shaking time and technique influence color development. Analyze a standard solution several times and adjust the shaking time to obtain the correct result. Use this time for analyzing samples					

## 1

#### **Procedures**

#### **Cadmium Reduction Method**



1. Fill a sample cell with 10 mL of sample. (the prepared sample)



Add the contents of one Nitrate MR
 Reagent Pillow to the cylinder. Stopper.
 Shake the sample cell vigorously for
 minutes. A 5 minute reaction period will begin. An amber color will develop if nitrate is present.



3. Fill another sample cell with 10 mL of sample. (the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 54.** 

(HACH DR/890 : 54

DR/2010 & 2500 : 353

DR/4000 : 2520)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



6. Within 2 minutes after the timer beeps, Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

Press Enter.

(Results will appear in mg/L NO<sub>3</sub>-N)

Nitrate, MR 48



## Nitrate, HR (0.3 $\sim$ 30.0 mg/L NO<sub>3</sub>-N)

## Cadmium Reduction Method

Required Reagents	Nitrate HR Rea	agent Pillow (Cadmium Reduction Method)	Cat. NO.	10424 - 11			
	Calcium 100 mg/L						
	Chloride	Concentrations Above 100 mg/L will caus a calibration must be done using standard					
	Ferric iron	At all levels					
Interferences	Nitrite	At all levels: This method measures both the nitrate and nitrite in the sample. If nitrite is present LR Test (Prog.# 60) should be done on the sample. Pretreat the nitrate nitrogen sample with the pretreatment. Then subtract the amount of nitrite found from the results of the NO <sub>2</sub> -N, LR Test; Add 30-g/L Bromine Water dropwise to the sample in step 2 until a yellow color remains. Mix after each drop. Add one drop of 30-g/L Phenol Solution to destroy the color.					
	рН	Highly buffered samples or extreme samp require sample pretreatment	nple pH may exceed the buffering capacity of the reagents and				
Sampling Storage & Preservation	More reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 48 hours at 4 °C. To preserve samples for longer periods, add 2mL of sulfuric acid per liter and store at 4°C. Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5N NaOH solution. Do not use mercury compounds as preservatives.						
Tips & Techniques	For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust. Rinse the sample cell and mixing cylinder immediately after use to remove all cadmium particles.  A deposit of unoxidized metal will remain after the Nitrate HR Reagent Pillow dissolves. The deposit will not affect results. Shaking time and technique influence color development. Analyze a standard solution several times and adjust the shaking time to obtain the correct result. Use this time for analyzing samples						

Nitrate, HR 49

## 1

#### **Procedures**

#### Cadmium Reduction Method



1. Fill a sample cell with 10 mL of sample. (the prepared sample)



Add the contents of one Nitrate HR
 Reagent Pillow to the cylinder. Stopper.
 Shake the sample cell vigorously for
 minutes. A 5 minute reaction period will begin. An amber color will develop if nitrate is present.



3. Fill another sample cell with 10 mL of sample. (the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 51**.

(HACH DR/890 : 51

DR/2010 & 2500 : 355

DR/4000 : 2530)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



6. Within 1 minutes after the timer beeps, Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

(Results will appear in mg/L NO<sub>3</sub>-N)

Nitrate, HR 50



## Nitrite, LR $(0.002\sim0.350$ mg/L $NO_2$ -N)

## **Diazotization Method**

Required Reagents	Nitrite LR Reagent Pillow	Cat. NO.	10512-00	
Interferences	Aluminous ions, Auric ions  Bismuth ions, Chloroplatinate ions  Ferric ions, Lead ions  Mercurous ions, Metavanadate ions  Silver ions	By causing precipitation		
	Cupric ions, Ferrous ions  Nitrate  Extreme sample pH or highly buffered samples	Cause low results  Above 100 mg/L as NO <sub>3</sub> -N  At all levels		
Sampling Storage & Preservation	Collect samples in clean plastic or glass bottles. Store at 4 °C (30 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. Do not use acid preservatives.			
Tips & Techniques	For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.			

#### **Procedures Diazotization Method**



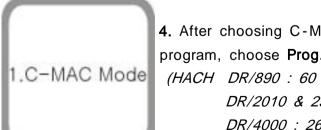
1. Fill a sample cell with 10 mL of sample. (the prepared sample)



2. Add the contents of one Nitrite LR Reagent Pillow. Cap and shake to dissolve. A pink color will develop if nitrite is present. A 20 minutes reaction period will begin.



3. Fill another sample cell with 10 mL of sample. (the blank)



4. After choosing C-MAC mode in the program, choose Prog.# 60.

DR/2010 & 2500 : 371

DR/4000 : 2610)



5. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



6. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

Press Enter.

(Results will appear in mg/L NO<sub>2</sub> - N)

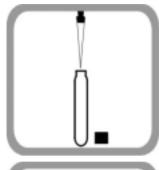


## Nitrite, LR $(0.003\sim0.500 \text{ mg/L NO}_2$ -N)

Diazotization Method ; TEST KIT

Required Reagents	Nitrite LR Vial	Cat. NO.	10512-01	
Interferences	Aluminous ions, Auric ions  Bismuth ions, Chloroplatinate ions  Ferric ions, Lead ions  Mercurous ions, Metavanadate ions  Silver ions  Cupric ions, Ferrous ions  Nitrate  Extreme sample pH or	By causing precipitation  Cause low results  Above 100 mg/L as NO <sub>3</sub> -N		
	highly buffered samples	At all levels		
Sampling Storage & Preservation	Collect samples in clean plastic or glass bottles. Store at 4 °C (30 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. Do not use acid preservatives.			
Tips & Techniques	For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results of perform a reagent blank adjust.			

#### Diazotization Method; TEST KIT



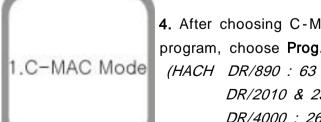
1. Fill a vial with 5 mL of sample. (the prepared sample)



2. Cap and shake to dissolve the powder. A pink color will develop if nitrite is present. A 20 minutes reaction period will begin.



3. Fill an empty vial with 5 mL of sample. (the blank)



4. After choosing C-MAC mode in the program, choose Prog.# 63.

DR/2010 & 2500 : 345

DR/4000 : 2630)



5. Wipe the blank and place it into the cell holder. Place the cover on the vial. Press Zero.



6. Wipe the prepared sample and place it into the cell holder. Place the cover on the vial. Press Enter. (Results will appear in mg/L NO<sub>2</sub> - N)



## Nitrite, HR ( $2\sim150$ mg/L $NO_2^-$ )

## Ferrous Sulfate Method

Required Reagents	Nitrite HR Reagent Pillow	Cat. NO.	10513-00		
Interferences	This test does not measure nitrates nor is it applicable to glycol-based samples.  Dilute glycol-based samples and follow the Low Range Nitrite procedure.				
Sampling, Storage & Preservation	Collect samples in clean plastic or glass bottles. The following storage instructions are necessary only when prompt analysis is impossible. Store at 4 °C (30 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. Do not use acid preservatives.				
Tips & Techniques	For more accurate results, determine a reagent blank value for each new lot of reagent Follow the procedure using deionized water in place of the sample.  Subtract the reagent blank value from the final results of perform a reagent blank adjusted.				

#### **Ferrous Sulfate Method**



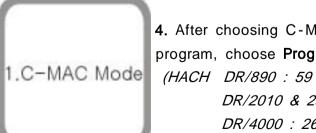
1. Fill a vial with 10 mL of sample. (the prepared sample)



2. Add the contents of one Nitrite HR Reagent Pillow. Cap and shake to dissolve. A 10 minutes reaction period will begin. Do not disturb it during the reaction period.



3. Fill another sample cell with 10 mL of sample. (the blank)



4. After choosing C-MAC mode in the program, choose Prog.# 59.

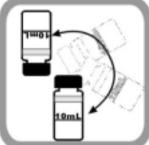
DR/2010 & 2500 : 373

DR/4000 : 2600)



5. Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



6. After the timer beeps, gently invert the prepared sample twice.

Avoid excessive mixing, or low results may occur.



7. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L NO<sub>2</sub>-)



## Nitrogen, Ammonia, LR $(0.02\sim2.50$ mg/L NH<sub>3</sub>-N)

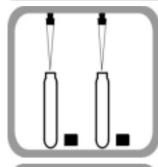
## Salicylate Method

Required Reagents	Ammonia Nitrogen LR Vial (Salicylate Method) Ammonia Reagent Pillow-1 Ammonia Reagent Pillow-2  Cat. NO. 10332-00					
	Calcium	2500 mg/L as CaCO <sub>3</sub>				
	Iron	Blank with ammonia free water	er of the same iron cond	centration.		
	Magnesium	15000 mg/L as CaCO₃				
	Nitrite	30 mg/L as NO <sub>2</sub> -N				
Interferences	Nitrate 250 mg/L as NO <sub>3</sub> -N					
	Orthophosphate	250 mg/L as PO <sub>4</sub> <sup>3-</sup> P				
	рН	Use 1N NaOH solution for acidic samples and 1N HCl solution for basic samples.				
	Sulfate	300 mg/L as $SO_4^{2-}$				
	Sulfide	Add the contents of one Sulfide Inhibitor Reagent Pillow. Swirl to mix. Filter.				
	Other	Hydrazine, glycine, turbidity, color : Distillate				
Sampling Storage & Preservation	Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1N Sodium thiosulfate for each $0.3 \text{mg/L Cl}_2$ in a 1L sample. Preserve the sample by reducing the pH to 2 or less with at least 2 mL of HCl. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize to pH 7 with 5N NaOH solution.					
Tips & Techniques	The ammonia salicylate reagent contains sodium nitroferricyanide. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas.					

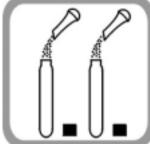
Nitrogen, Ammonia, LR 57

### Salicylate Method

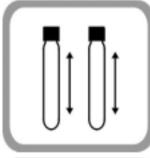
## Procedures



Add 2 mL of sample to one Ammonia
 Nitrogen LR Vial. (the prepared sample)
 Add 2 mL of deionized water to another vial. (the blank)



**2.** Add the contents of Ammonia Reagent 1 Pillow to each vial.



3. Cap vials and shake to dissolve.

Add the contents of Ammonia Reagent 2

Pillow to each vial. Cap vials and shake to dissolve. A 20 minute reaction period will begin. A green color will develop if ammonia is present.



**4.** After choosing C-MAC mode in the program, choose **Prog.# 66.** 

(HACH DR/890 : 66

DR/2010 & 2500 : 342

DR/4000 : 2460)



**5.** Wipe the blank and place it into the cell holder. Place the cover on the vial. Press Zero.



**6.** Wipe the prepared sample and place it into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L NH<sub>3</sub>-N)

Nitrogen, Ammonia, LR 58



## Nitrogen, Ammonia, HR (0.4 $\sim$ 50.0 mg/L NH<sub>3</sub>-N)

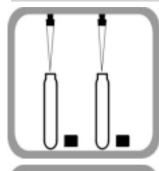
## Salicylate Method

Required Reagents	Ammonia Nitrogen HR Vial (Salicylate Method) Ammonia Reagent Pillow-1 Ammonia Reagent Pillow-2  Cat. NO. 10333-00				
	Calcium Iron Magnesium Nitrite	50,000 mg/L as CaCO <sub>3</sub> Blank with ammonia free water of the same iron concentration.  300,000 mg/L as CaCO <sub>3</sub> 600 mg/L as NO <sub>2</sub> -N			
Interferences	Nitrate Orthophosphate pH Sulfate	$5,000 \text{ mg/L}$ as $NO_3$ -N $5,000 \text{ mg/L}$ as $PO_4^3$ -P Use 1N NaOH solution for acidic samples and 1N HCl solution for basic samples. $6,000 \text{ mg/L}$ as $SO_4^2$ -			
	Sulfide Other	Add the contents of one Sulfide Inhibitor Reagent Pillow. Swirl to mix. Filter.  Hydrazine, glycine, turbidity, color: Distillate			
Sampling Storage & Preservation	Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1N Sodium thiosulfate for each $0.3 \text{mg/L Cl}_2$ in a 1L sample. Preserve the sample by reducing the pH to 2 or less with at least 2 mL of HCl. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize to pH 7 with 5N NaOH solution.				
Tips & Techniques	The ammonia salicylate reagent contains sodium nitroferricyanide. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas.				

Nitrogen, Ammonia, HR 59

### Salicylate Method

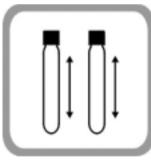
## Procedures



 Add 0.1 mL of sample to one Ammonia Nitrogen HR Vial. (the prepared sample)
 Add 0.1mL of deionized water to another vial. (the blank)



**2.** Add the contents of Ammonia Reagent 1 Pillow to each vial.



3. Cap vials and shake to dissolve.

Add the contents of Ammonia Reagent 2

Pillow to each vial. Cap vials and shake to dissolve. A 20 minute reaction period will begin. A green color will develop if ammonia is present.



**4.** After choosing C-MAC mode in the program, choose **Prog.# 67.** 

(HACH DR/890 : 67

DR/2010 & 2500 : 343

DR/4000 : 2465)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the vial. Press Zero.



**6.** Wipe the prepared sample and place it into the cell holder. Place the cover on the vial.

Press Enter.

(Results will appear in mg/L NH<sub>3</sub>-N)

Nitrogen, Ammonia, HR 60



## Nitrogen, Total (TN), LR (3.0 $\sim$ 25.0 mg/L N)

## Chromotropic Acid Method

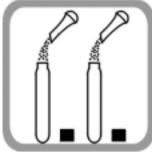
Required Reagents	Total Nitroger Total Nitroger Total Nitroger	Hydroxide Vial Persulfate Reagent Pillow Acid Solution Vial Reagent 1 Pillow Reagent 2 Pillow	Cat. NO.	10212-00	
	Barium	>2.6 mg/L	Magnesium	>500 mg/L	
	Bromide	>60 mg/L : Positive	Organic carbon	>150 mg/L	
	Calcium	>300 mg/L	рН	>13	
Interferences	Chloride	>1000 mg/L : Positive	Phosphorus	>100 mg/L	
	Chromium( <sup>3+</sup> )	>0.5 mg/L	Silica	>150 mg/L	
	Iron	>2 mg/L	Silver	>0.9 mg/L	
	Lead	>6.6µg/L	Tin	>1.5 mg/L	
Sampling Storage & Preservation	analysis. Adjust sample pH to 2 or less with Concentrated Sulfuric Acid (about 2 mL per liter). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days.  Warm the samples to room temperature and neutralize with 5N NaOH solution before				
Tips & Techniques	This test is technique-sensitive. Invert the vials as described here to avoid low results: Hold the vial in a vertical position with the cap pointing up. Turn the vial upside-down. Wait for all of the solution to flow down to the cap. Pause. Return the vial to an upright position. Wait for all the solution to flow to the bottom of the vial. This process equals one inversion.				

Nitrogen, Total (TN), LR 61

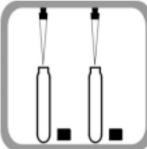
### Property

#### **Procedures**

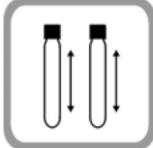
### Chromotropic Acid Method



1. Add the contents of Total Nitrogen-Persulfate Reagent Pillow to each Total Nitrogen Hydroxide Vial. Wipe off any reagent that may get on the lid or the vial threads.



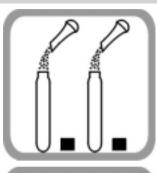
2. Add 2 mL of sample to one Ammonia Nitrogen LR Vial. (the prepared sample) Add 2 mL of deionized water to another vial. (the blank)



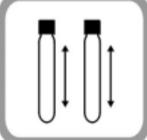
3. Cap vials and shake to dissolve vigorously for at least 30 seconds to mix. The reagent may not dissolve completely after shaking. This will not affect accuracy.



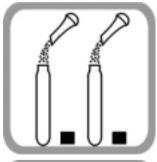
4. Place the vials in the reactor preheated to 105. Heat for exactly 30 minutes.Place the hot vials into a rack from the reactor. Cool the vials to room temperature.



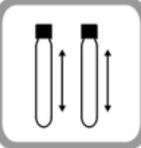
**5.** Remove the caps from the digested vials and add the contents of one Total Nitrogen Reagent 1 Pillow to each vials.



6. Cap the vials and shake for 15 seconds. A 3 minute reaction period will begin.



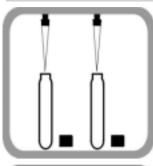
7. Add the contents of one TotalNitrogen Reagent 2 Pillow to each vials.



**8.** Cap the vials and shake for 15 seconds. A **2 minute** reaction period will begin. The reagent may not dissolve completely after shaking. This will not affect accuracy. The solution will begin to turn light yellow.

Nitrogen, Total (TN), LR 62

### Chromotropic Acid Method

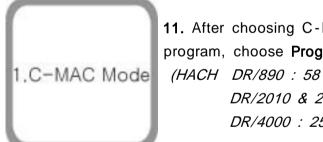


9. Remove the caps from two vials and add 2 mL of digested, treated sample to one Total Nitrogen Acid Solution Vial. (the prepared sample). Add 2 mL of digested, treated reagent blank to the second Total Nitrogen Acid Solution Vial. (the blank)



10. Cap vials and invert 10 times to mix. Use slow, deliberate inversions for complete recovery. The vials will be warm.

A 5 minute reaction period will begin. The yellow color will intensify.



11. After choosing C-MAC mode in the program, choose Prog.# 58.

DR/2010 & 2500 : 350

DR/4000 : 2558)



12. Wipe the blank and place it into the cell holder.

Place the cover on the vial.

Press Zero.



13. Wipe the prepared sample and place it into the cell holder.

Place the cover on the vial.

Press Enter.

(Results will appear in mg/L N)

63 Nitrogen, Total (TN), LR



## Nitrogen, Total (TN), HR ( $10\sim150$ mg/L N)

## Chromotropic Acid Method

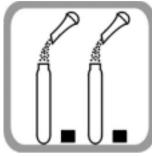
Required Reagents	Total Nitroger Total Nitroger Total Nitroger	Hydroxide Vial Persulfate Reagent Pillow Acid Solution Vial Reagent 1 Pillow Reagent 2 Pillow	Cat. NO.	10213-00	
	Barium	>10 mg/L	Magnesium	>2000 mg/L	
	Bromide	>240 mg/L : Positive	Organic carbon	>600 mg/L	
	Calcium	>1200 mg/L	рН	>13	
Interferences	Chloride	>3000 mg/L : Positive	Phosphorus	>400 mg/L	
	Chromium(3+)	>2 mg/L	Silica	>600 mg/L	
	Iron	>8 mg/L	Silver	>3 mg/L	
	Lead	>26µg/L	Tin	>6 mg/L	
Sampling Storage & Preservation	Storage  Storage  liter). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days.  Warm the samples to room temperature and neutralize with 5N NaOH solution before				
Tips & Techniques	This test is technique-sensitive. Invert the vials as described here to avoid low results: Hold the vial in a vertical position with the cap pointing up. Turn the vial upside-down. Wait for all of the solution to flow down to the cap. Pause. Return the vial to an upright position. Wait for all the solution to flow to the bottom of the vial. This process equals one inversion.				

Nitrogen, Total (TN), HR 65

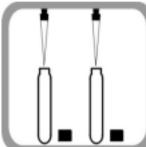
## Pro

#### **Procedures**

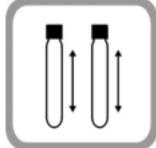
### Chromotropic Acid Method



1. Add the contents of Total Nitrogen-Persulfate Reagent Pillow to each Total Nitrogen Hydroxide Vial. Wipe off any reagent that may get on the lid or the vial threads.



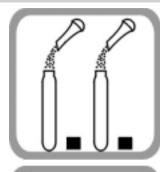
 Add 0.5 mL of sample to one Ammonia Nitrogen LR Vial. (the prepared sample)
 Add 0.5 mL of deionized water to another vial. (the blank)



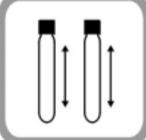
3. Cap vials and shake to dissolve vigorously for at least 30 seconds to mix. The reagent may not dissolve completely after shaking. This will not affect accuracy.



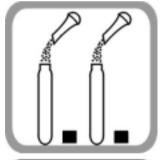
4. Place the vials in the reactor preheated to 105. Heat for exactly 30 minutes.Place the hot vials into a rack from the reactor. Cool the vials to room temperature.



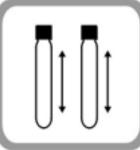
**5.** Remove the caps from the digested vials and add the contents of one Total Nitrogen Reagent 1 Pillow to each vials.



6. Cap the vials and shake for 15 seconds. A 3 minute reaction period will begin.



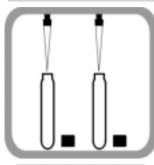
7. Add the contents of one TotalNitrogen Reagent 2 Pillow to each vials.



**8.** Cap the vials and shake for 15 seconds. A **2 minute** reaction period will begin. The reagent may not dissolve completely after shaking. This will not affect accuracy. The solution will begin to turn light yellow.

Nitrogen, Total (TN), HR 66

### Chromotropic Acid Method

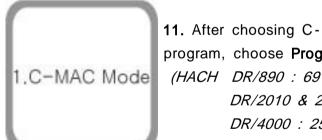


9. Remove the caps from two vials and add 2 mL of digested, treated sample to one Total Nitrogen Acid Solution Vial. (the prepared sample). Add 2 mL of digested, treated reagent blank to the second Total Nitrogen Acid Solution Vial. (the blank)



10. Cap vials and invert 10 times to mix. Use slow, deliberate inversions for complete recovery. The vials will be warm.

A 5 minute reaction period will begin. The yellow color will intensify.



11. After choosing C-MAC mode in the program, choose Prog.# 69.

DR/2010 & 2500 : 395

DR/4000 : 2559)



12. Wipe the blank and place it into the cell holder.

Place the cover on the vial.

Press Zero.



13. Wipe the prepared sample and place it into the cell holder.

Place the cover on the vial.

Press Enter.

(Results will appear in mg/L N)

*67* Nitrogen, Total (TN), HR



# Oxygen Demand, Chemical (COD $_{\text{Cr}}$ ) ULR (2 $^{\sim}$ 40 mg/L COD)

## Reactor Digestion Method

Required Reagents	COD ULR Vial		Cat. NO.	10111-00		
	Chloride is the primary interference when determining COD concentration. Samples with higher chloride concentrations should be diluted. If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.5 g of mercuric sulfate to each COD vial before the sample is added.					
Interference		Maximum Cl <sup>-</sup> (mg/L)	Suggested Cl <sup>-</sup> of diluted samples (mg/L)		When 0.5g HgSO₄ added Maximum Cl⁻(mg/L)	
	ULR	2000	1000		NA	
	LR,HR	2000	1000		LR: 8000, HR: 4000	
	UHR	20,000	10,000		40,000	
Sampling Storage & Preservation	Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days.					
Tips & Techniques	Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water.  Place a safety shield in front of the COD reactor to prevent injury if splattering occurs.  The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container. Refrigerate if possible. Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Wash spills with running water. Run one blank with each set of samples. Run all tests (the samples and the blank) with the same lot of vials. For greater accuracy, analyze a minimum of three replicates and average the results.					

## **Reactor Digestion Method**



## **Procedures**



Hold one vial at a 45 degree angle.
 Add 2 mL of sample to vial.
 (the prepared sample)
 Hold one vial at a 45 degree angle.
 Add 2 mL of deionized water to vial.
 (the blank)



2. Cap the vials tightly. Rinse them with deionized water and wipe with a clean paper towel. Hold the vials by the cap over a sink. Invert gently several times to mix. The samples vials will becoming very hot during mixing.



Place the vials in the COD reactor preheated to 150 . Heat for 2 hours.
 Turn the reactor off. Wait about 20 minutes for the vials to cool to 120°C or less.



**4.** Invert each vials several times while still warm. Place the vials into a rack and cool to room temperature.



**5.** After choosing C-MAC mode in the program, choose **Prog.# 12**.

(HACH DR/2010 & 2500 : 431 DR/4000 : 2700)



**6.** Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.



7. Place the blank into the cell holder.Place the cover on the vial.Press Zero.



**8.** Place prepared sample into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L COD)



# Oxygen Demand, Chemical (COD $_{cr}$ ) LR (10 $\sim$ 150 mg/L COD)

# **Reactor Digestion Method**

Required Reagents	COD LR Vial		Cat. NO.	10112-00				
	Chloride is the primary interference when determining COD concentration. Samples with higher chloride concentrations should be diluted. If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.5 g of mercuric sulfate to each COD vial before the sample is added.							
Interference		Maximum CI <sup>-</sup> (mg/L)	Cl <sup>-</sup> of s (mg/L)	When 0.5g HgSO₄ added Maximum Cl⁻(mg/L)				
	ULR	2000	1000		NA			
	LR,HR	2000	1000		LR: 8000, HR: 4000			
	UHR	20,000	10,000		40,000			
Sampling Storage & Preservation	Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination.  Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter)  and refrigerated at 4 °C can be stored up to 28 days.							
Tips & Techniques	Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water.  Place a safety shield in front of the COD reactor to prevent injury if splattering occurs.  The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container. Refrigerate if possible.							

## Reactor Digestion Method



1. Hold one vial at a 45 degree angle. Add 2 mL of sample to vial. (the prepared sample) Hold one vial at a 45 degree angle. Add 2 mL of deionized water to vial. (the blank)



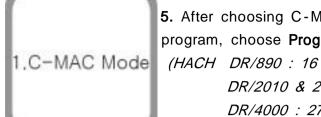
2. Cap the vials tightly. Rinse them with deionized water and wipe with a clean paper towel. Hold the vials by the cap over a sink. Invert gently several times to mix. The samples vials will becoming very hot during mixing.



3. Place the vials in the COD reactor. preheated to 150 . Heat for 2 hours. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120°C or less.



4. Invert each vials several times while still warm. Place the vials into a rack and cool to room temperature.



5. After choosing C-MAC mode in the program, choose Prog.# 16.

DR/2010 & 2500 : 430

DR/4000 : 2710)



6. Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.



7. Place the blank into the cell holder. Place the cover on the vial. Press Zero.



8. Place prepared sample into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L COD)



# Oxygen Demand, Chemical (COD $_{\text{Cr}}$ ) HR (100 $^{\sim}$ 1500 mg/L COD)

# Reactor Digestion Method

Required Reagents	COD HR Vial		Cat. NO.	10113-00				
	Chloride is the primary interference when determining COD concentration. Samples with higher chloride concentrations should be diluted. If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.5 g of mercuric sulfate to each COD vial before the sample is added.							
Interference		When 0.5g HgSO₄ added Maximum Cl⁻(mg/L)						
	ULR	2000	1000 1000 10,000		NA			
	LR,HR	2000			LR: 8000, HR: 4000			
	UHR	20,000			40,000			
Sampling Storage & Preservation	Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination.  Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days.							
Tips & Techniques	Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water.  Place a safety shield in front of the COD reactor to prevent injury if splattering occurs.  The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container. Refrigerate if possible.							

## Reactor Digestion Method



1. Hold one vial at a 45 degree angle. Add 2 mL of sample to vial. (the prepared sample) Hold one vial at a 45 degree angle. Add 2 mL of deionized water to vial. (the blank)



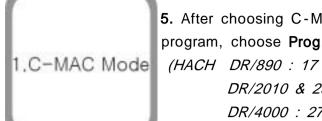
2. Cap the vials tightly. Rinse them with deionized water and wipe with a clean paper towel. Hold the vials by the cap over a sink. Invert gently several times to mix. The samples vials will becoming very hot during mixing.



3. Place the vials in the COD reactor. preheated to 150 . Heat for 2 hours. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120°C or less.



4. Invert each vials several times while still warm. Place the vials into a rack and cool to room temperature.



5. After choosing C-MAC mode in the program, choose Prog.# 17.

DR/2010 & 2500 : 435

DR/4000 : 2720)



6. Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.



7. Place the blank into the cell holder. Place the cover on the vial. Press Zero.



8. Place prepared sample into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L COD)



# Oxygen Demand, Chemical (COD $_{cr}$ ) UHR (1000 $\sim$ 15000 mg/L COD)

# **Reactor Digestion Method**

Required Reagents	COD HR Vial		Cat. NO.	10113-00				
	Chloride is the primary interference when determining COD concentration. Samples with higher chloride concentrations should be diluted. If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.5 g of mercuric sulfate to each COD vial before the sample is added.							
Interference		Maximum Cl <sup>-</sup> (mg/L)	When 0.5g HgSO₄ added Maximum Cl⁻(mg/L)					
	ULR	2000	1000		NA			
	LR,HR	2000	1000		LR: 8000, HR: 4000			
	UHR	20,000	10,000		40,000			
Sampling Storage & Preservation	Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination.  Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter)  and refrigerated at 4 °C can be stored up to 28 days.							
Tips & Techniques	Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water.  Place a safety shield in front of the COD reactor to prevent injury if splattering occurs.  The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container. Refrigerate if possible.							

## Reactor Digestion Method



1. Hold one vial at a 45 degree angle. Add 0.2 mL of sample to vial. (the prepared sample) Hold one vial at a 45 degree angle. Add 0.2 mL of deionized water to vial. (the blank)



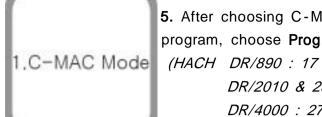
2. Cap the vials tightly. Rinse them with deionized water and wipe with a clean paper towel. Hold the vials by the cap over a sink. Invert gently several times to mix. The samples vials will becoming very hot during mixing.



3. Place the vials in the COD reactor. preheated to 150 . Heat for 2 hours. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120°C or less.



4. Invert each vials several times while still warm. Place the vials into a rack and cool to room temperature.



5. After choosing C-MAC mode in the program, choose Prog.# 17.

DR/2010 & 2500 : 435

DR/4000 : 2720)



6. Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.



7. Place the blank into the cell holder. Place the cover on the vial. Press Zero.



8. Place prepared sample into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L COD) Multiply the result by 10.



Phosphorus, Reactive, LR  $(0.06\sim5.00 \text{ mg/L PO}_4^{3-} / 0.02\sim1.60 \text{ mg/L P})$ 

**Ascorbic Acid Method** 

Required Reagents	Phosphorus Vial PO <sub>4</sub> -P LR Reagent Pillow		Cat. NO.	10712-00			
	Aluminum	Greater than 200	) mg/L				
	Arsenate	At all levels					
	Chromium	Greater than 100	) mg/L				
	Copper, Silicate	Greater than 10	mg/L				
	Iron	Greater than 100	) mg/L				
	Nickel	Greater than 300	) mg/L				
Interferences	Highly buffered samples or extreme pH	May exceed the buffering capacity of the reagents and require sample pretreatment.					
	Silica	Greater than 50 mg/L					
	Sulfide	Greater than 6 mg/L: Swirling constantly 25mL of sample, add bromine water drop-wise until a permanent yellow color appears. Add phenol solution drop-wise until the yellow color disappears.					
	Turbidity or color	May cause inconsistent results.					
	Zinc	Greater than 80 mg/L					
Sampling Storage & Preservation	Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 HCl and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test. Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 48 hours by filtering immediately and storing at 4 °C. Warm samples to room temperature before analysis.						
Tips & Techniques	Store the PO <sub>4</sub> -P LR reagent pillows in a cool, dry environment.						

1

#### **Procedures**

1. After choosing C-MAC mode in the

program, choose **Prog.# 82**. 1.C-MAC Mode *(HACH DR/890 : 82* 

DR/2010 & 2500 : 535

DR/4000 : 3035)



2. Add 5 mL of sample to a vial. Cap and mix.



**3.** Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks..



**4.** Wipe the blank and place it into the cell holder.

Place the cover on the vial.

Press Zero.



**5.** Add the contents of one PO<sub>4</sub>-P LR Reagent Pillow to the vial. Cap and shake for 10 ~ 15 seconds. The powder will not dissolve completely. A **2 minute** reaction period will begin. Read samples between 2 and 8 minutes after adding the reagent.

**Ascorbic Acid Method** 



**6.**Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.



7. Wipe the prepared sample and place it into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L P) Generally Chemical form of hach spectrophotometer is  $PO_4^{3-}$ .



# Phosphorus, Reactive, HR $(1.0\sim100 \text{ mg/L PO}_4^{3-} / 0.4\sim30.0 \text{ mg/L P})$

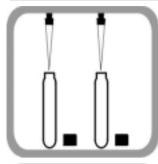
# **Molybdovanadate Method**

Required Reagents	Phosphorus Vial TP Solution 2	<b>Cat. NO.</b> 10723-00					
	Arsenate	Only interferes if the sample is heated					
	Iron, ferrous	Above 100 mg/L					
	Molybdate	Above 1000 mg/L : negative inteference					
	Silica	Only interferes if the sample is heated					
Interferences	Highly buffered samples or extreme pH	May exceed the buffering capacity of the reagents and require sample pretreatment.					
	Temperature	Less than 18 , Greater than 25					
		Fluoride, thorium, bismuth, thiosulfate or thiocyanate					
	Other	Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, salicylate,Al <sup>3+</sup> ,Fe <sup>3+</sup> ,Mg <sup>2+</sup> ,Ca <sup>2+</sup> ,Ba <sup>2+</sup> ,Sr <sup>2+</sup> ,Li <sup>+</sup> ,Na <sup>+</sup> ,K <sup>+</sup> ,NH <sub>4</sub> <sup>+</sup> , Cd <sup>2+</sup> ,Mn <sup>2+</sup> ,NO <sub>3</sub> <sup>-</sup> ,NO <sub>2</sub> <sup>-</sup> ,SO <sub>4</sub> <sup>2-</sup> ,SO <sub>3</sub> <sup>2-</sup> ,Pb <sup>2+</sup> ,Hg <sup>+</sup> ,Hg <sup>2+</sup> ,Sn <sup>2+</sup> ,Cu <sup>2+</sup> ,Ni <sup>2+</sup> ,Ag <sup>+</sup> ,U <sup>4+</sup> , Zn <sup>4+</sup> , AsO <sub>3</sub> <sup>-</sup> , Br <sup>-</sup> ,CO <sub>3</sub> <sup>2-</sup> ,ClO <sub>4</sub> <sup>-</sup> ,CN <sup>-</sup> ,lO <sub>3</sub> <sup>-</sup> ,SlO <sub>4</sub> <sup>4-</sup> : Above 1000 mg/L					
Sampling Storage & Preservation	with deionized water. Do glassware used in this tanalysis is impossible, s	ic or glass bottles that have been acid cleaned with 1:1 HCl and rinsed on not use commercial detergents containing phosphate for cleaning test. Analyze samples immediately after collection for best results. If proresamples may be preserved up to 28 days by adjusting the pH to 2 or lest 2 mL per liter) and storing at 4 °C. Warm samples to room temperature NaOH before analysis.					

## Molybdovanadate Method



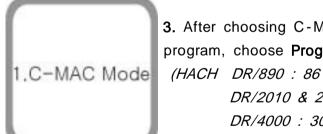
#### **Procedures**



1. Add 5 mL of sample to one Ammonia Nitrogen HR Vial. (the prepared sample) Add 5 mL of deionized water to another vial. (the blank)



2. Cap and mix. A 3 minute reaction period will begin. (A 7 minute reaction time is for samples at 23 °C. For samples at 13 °C, wait 15 minutes. For samples at 33 °C, wait 2 minutes.) Read the sample within 2 minutes after the timer beeps.



3. After choosing C-MAC mode in the program, choose Prog.# 86.

DR/2010 & 2500 : 540

DR/4000 : 3000)



4. Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.



5. Wipe the blank and place it into the cell holder.

Place the cover on the vial. Press Zero.



6. Wipe the prepared sample and place it into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L P) Generally Chemical form of hach spectrophotometer is  $PO_4^{3-}$ .



# Phosphorus, Total, LR (0.06 $\sim$ 3.50 mg/L PO $_4^{3-}$ / 0.02 $\sim$ 1.10 mg/L P )

# Acid Persulfate Method

Required Reagents	TP Vial TP Solution 1 TP Persulfate Reagent Pillow TP LR Reagent Pillow		Cat. NO.	10612-00		
	Aluminum	Greater than 200 mg/L	Nickel	Greater than 300 mg/L		
	Arsenate	At all levels	Silica	Greater than 50 mg/L		
	Chromium	Greater than 100 mg/L	Turbidity	May cause inconsistent results.		
	Copper, Silicate	Greater than 10 mg/L	Zinc	Greater than 80mg/L		
Interferences	Iron	Greater than 100 mg/L				
	Highly buffered samples or extreme pH	May exceed the buffering capacity of the reagents and require sample pretreatment.				
	Sulfide	Greater than 6 mg/L: Swirling constantly 25mL of sample, add bromine water drop-wise until a permanent yellow color appears. Add phenol solution drop-wise until the yellow color disappears.				
Sampling Storage & Preservation  Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 HCl and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.  Analyze samples immediately after collection for best results. If prompt analysis is impossible, samples may be preserved up to 28 days by adjusting the pH to 2 or less with sulfuric acid (about 2 mL per liter) and storing at 4 °C. Warm samples to room temperature and neutralize with 5N NaOH before analysis.						
Tips & Techniques	Place a safety shield in front of the COD reactor to prevent injury if splattering occurs.					

# 1

## **Procedures**

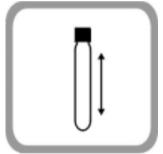
#### Acid Persulfate Method



1. Add 5 mL of sample to a vial.



**2.** Add the contents of one TP Persulfate Reagent Pillow to the vial.



3. Cap tightly and shake to dissolve.



4. Place the vials in the reactor preheated to 120. Heat for exactly 30 minutes.Place the hot vials into a rack from the reactor. Cool the vials to room temperature.



5. Add 2 mL of TP Solution-1 to the vial.



6. Cap and invert to mix.



**7.** After choosing C-MAC mode in the program, choose **Prog.# 82.** 

(HACH DR/890 : 82

DR/2010 : 535

DR/2500 : 536

DR/4000 : 3036)



**8.** Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.

# Acid Persulfate Method

Procedures



**9.** Wipe the blank and place it into the cell holder.

Place the cover on the vial. Press Zero.



10. Add the contents of one TP LR Reagent Pillow to the vial. Cap and shake for 10 ~ 15 seconds. The powder will not dissolve completely. A 2 minute reaction period will begin. Read samples between 2 and 8 minutes after adding the reagent.



**11.** Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks..



**12.** Wipe the prepared sample and place it into the cell holder. Place the cover on the vial. Press Enter.

(Results will appear in mg/L P) Generally Chemical form of hach spectrophotometer is  $PO_4^{3-}$ .



# Phosphorus, Total, HR

# Molybdovanadate

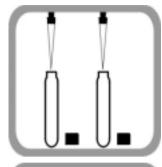
 $(1.0 \sim 100 \text{ mg/L PO}_4^{3-} / 0.4 \sim 30.0 \text{ mg/L P})$  Method

Required Reagents	TP Vial TP Solution 1 TP Persulfate Reagent Pillow TP Solution 2		Cat. NO.	10623-00		
	Arsenate	Only interferes if the	ne sample is heated	1		
	Iron, ferrous	Above 100 mg/L				
	Molybdate	Above 1000 mg/L	: negative inteferen	ce		
	Silica Only interferes if the sample is heated					
Interferences	Highly buffered samples or extreme pH	May exceed the buffering capacity of the reagents and require sample pretreatment.				
interrerences	Temperature	Less than 18 , Greater than 25				
		Fluoride, thorium, bismuth, thiosulfate or thiocyanate				
	Other	Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, salicylate,Al <sup>3+</sup> ,Fe <sup>3+</sup> ,Mg <sup>2+</sup> ,Ca <sup>2+</sup> ,Ba <sup>2+</sup> ,Sr <sup>2+</sup> ,Li <sup>+</sup> ,Na <sup>+</sup> ,K <sup>+</sup> ,NH <sub>4</sub> <sup>+</sup> , Cd <sup>2+</sup> ,Mn <sup>2+</sup> ,NO <sub>3</sub> <sup>-</sup> ,NO <sub>2</sub> <sup>-</sup> ,SO <sub>4</sub> <sup>2-</sup> ,SO <sub>3</sub> <sup>2-</sup> ,Pb <sup>2+</sup> ,Hg <sup>+</sup> ,Hg <sup>2+</sup> ,Sn <sup>2+</sup> ,Cu <sup>2+</sup> ,Ni <sup>2+</sup> ,Ag <sup>+</sup> ,U <sup>4+</sup> , Zn <sup>4+</sup> , AsO <sub>3</sub> <sup>-</sup> , Br <sup>-</sup> ,CO <sub>3</sub> <sup>2-</sup> ,ClO <sub>4</sub> <sup>-</sup> ,CN <sup>-</sup> ,lO <sub>3</sub> <sup>-</sup> ,SlO <sub>4</sub> <sup>4-</sup> : Above 1000 mg/L				
Sampling Storage & Preservation	Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 HCl and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test. Analyze samples immediately after collection for best results. If prompt analysis is impossible, samples may be preserved up to 28 days by adjusting the pH to 2 or less with sulfuric acid (about 2 mL per liter) and storing at 4 °C. Warm samples to room temperature and neutralize with 5N NaOH before analysis.					

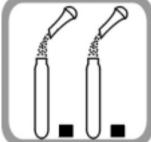
Phosphorus, Total (TP), HR

# Molybdovanadate Method

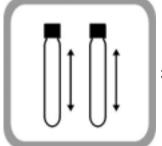
# Procedures



 Add 5 mL of sample to one Ammonia Nitrogen HR Vial. (the prepared sample)
 Add 5 mL of deionized water to another vial. (the blank)



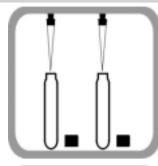
**2.** Add the contents of TP Persulfate Reagent Pillow to each vial.



3. Cap and mix to dissolve.



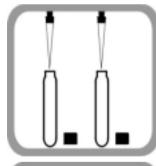
4. Place the vials in the reactor preheated to 120. Heat for exactly 30 minutes.Place the hot vials into a rack from the reactor. Cool the vials to room temperature.



**5.** Add **2 mL of TP Solution-1** to each vials.



6. Cap and invert to mix.



7. Add 0.5 mL of TP Solution-2 to each vials.



8. Cap and invert to mix.

A **7 minute** reaction period will begin. Read samples between 7 and 9 minutes after adding the TP Solution-2.



9. After choosing C-MAC mode in the program, choose Prog.# 87.

DR/2010 & 2500 : 541

DR/4000 : 3040)



10. Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks...



11. Wipe the blank and place it into the cell holder.

Place the cover on the vial. Press Zero.



12. Wipe the prepared sample and place it into the cell holder. Place the cover on the sample cell. Press Enter. (Results will appear in mg/L P) Generally Chemical form of hach spectrophotometer is PO<sub>4</sub><sup>3-</sup>.



# Silica (1.0 $\sim$ 75.0 mg/L SiO<sub>2</sub>)

# Silicomolybdate Method

Required Reagents	Acid Reagent Pillow for Silica Citric Acid Pillow Molybdate Reagent Pillow		Cat. NO.	12710-00		
	Color, Turbidity	Eliminated by zeroing	the instrument w	ith the original sample.		
	Iron	High levels of Fe <sup>2+</sup> an	d Fe <sup>3+</sup> interfere.			
Interferences	Phosphate	>60 mg/L PO <sub>4</sub> <sup>3-</sup> : a negative 2% interference occurs. >75 mg/L PO <sub>4</sub> <sup>3-</sup> : a negative 11% interference occurs.				
	Sulfides	At all levels				
Sampling Storage & Preservation	Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples at 4 °C (39 °F) for up to 28 days. Warm samples to room temperature before analyzing.					
Tips & Techniques	Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with Sodium Bicarbonate, then Sulfuric Acid will make these forms reactive to molybdate. The pretreatment is given in Standard Methods for the Examination of Water and Wastewater under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help instead of the bicarbonate treatment.  Sample temperature should be 15 ~ 25 °C (59 ~ 77 °F)					

Silica 89

## Silicomolybdate Method



1. Fill a sample cell with 10 mL of sample. (the prepared sample)



2. Add the contents of one Molybdate Reagent Pillow to the sample cell. Swirl until completely dissolved.



3. Add the contents of one Acid Reagent Pillow for Silica to the sample cell. Swirl to mix. A yellow color will develop if silica or phosphorus is present.

A 10 minute reaction period will begin.

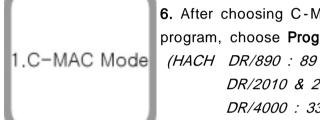


4. Add the contents of one Citric Acid Pillow for Silica to the sample cell. Swirl to mix.

A 2 minute reaction period will begin. Any yellow color due to phosphorus is removed in this step.



5. Fill a second sample cell with 10 mL of the original sample. (the blank)



6. After choosing C-MAC mode in the program, choose Prog.# 89.

DR/2010 & 2500 : 656

DR/4000 : 3350)



7. Within 3 minutes after the timer beeps, wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



8. Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L SiO<sub>2</sub>)

90 Silica



# Sulfate (2 $\sim$ 70 mg/L SO<sub>4</sub> $^{2-}$ )

# Sulfate Method

Required Reagents	Sulfate reagent pillow		Cat.	NO.	13010-00	
	Calcium	20,000 mg/L as CaCO <sub>3</sub>				
Interferences	Chloride	40,000 mg/L as	: CI			
interreterices	Magnesium	10,000 mg/L as	CaCO₃			
	Silica	500 mg/L as SIO <sub>2</sub>				
Sampling Storage & Preservation	Collect samples in clean plastic or glass bottles. Samples may be stored up to 7 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.					
Tips & Techniques	For best results, perform a new calibration for each lot of reagent.  For more accurate results, determine a reagent blank value for each new lot of reagent.  Follow the procedure using deionized water in place of the sample.  Subtract the reagent blank value from the final results or perform a reagent blank adjust.  Filter highly colored or turbid samples using filter paper and a funnel.  Undissolved powder that has settled does not affect accuracy.					

Sulfate 91

# Ŧ

# Procedures Sulfate Method



1. Fill a sample cell with 10 mL of sample. (the prepared sample)



2. Add the contents of one Sulfate Reagent Pillow to the sample cell. Swirl to mix.A 5 minute reaction period will begin.Do not disturb the cell during this time.



3. Fill a second sample cell with 10 mL of sample. (the blank)



**4.** After choosing C-MAC mode in the program, choose **Prog.# 91.** 

(HACH DR/890 : 91

DR/2010 & 2500 : 680

DR/4000 : 3450)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**6.** Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

Press Enter.

(Results will appear in mg/L SO<sub>4</sub><sup>2</sup>-)



**7.** Clean the sample cells with soap and a brush.

Sulfate 92



# Sulfide $(0.005 \sim 0.700 \text{ mg/L S}^2)$

# Methylene Blue Method

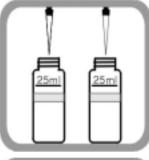
Required Reagents	Sulfide Solution 1 Sulfide Solution 2		Cat. NO.	13410-00			
	Strong reducing Sulfite, thiosulfate, hydrosulfite etc : by reducing the blue substances or its development						
	Sulfide, high levels	High concentrations of sulfite may inhibit full color development and sample dilution. Some sulfide loss may occur when the sample is diluted.					
Interferences	Turbidity	For turbid samples, prepare a sulide-free blank as follows. Use it in place of the deionized water blank in the procedure.  1. Measure 25 mL of sample into a 50-mL Erlenmeyer flask.  2. Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears.  3. Add Phenol Solution dropwise until the yellow color just disappears. Use this solution to replace the deionized water.					
Sampling, Storage & Preservation	Collect samples in clean plastic or glass bottles. Fill completely and cap tightly.  Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.						
Tips & Techniques	Analyze samples immediately. Do not preserve for later analysis. Avoid excessive agitation of samples to minimize sulfide loss. Some sulfide loss may occur if dilution is necessary. Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.						

Sulfate 93

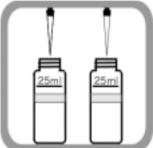
# 1

#### **Procedures**

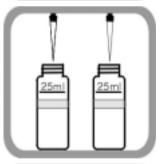
# Methylene Blue Method



Avoid excessive agitation of the sample, use a pipet add 25 mL of sample to a sample cell.(the prepared sample) use a pipet add 25 mL of deionized water to a second sample cell.(the blank)



2. Add 1 mL of Sulfide Solution-1 reagent to each cell. Swirl to mix.



3. Add 1 mL of Sulfide Solution-2 reagent to each cell. Cap and immediately inver to mix. A 5 minute reaction period will begin.



**4.** After choosing C-MAC mode in the program, choose **Prog.# 93**.

(HACH DR/890: 93

DR/2010 & 2500 : 690

DR/4000 : 3500)



**5.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**6.** Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell.

Press Enter.

(Results will appear in  $mg/L S^{2-}$ )

Sulfide 94



# Zinc ( $0.01\sim3.00$ mg/L Zn)

# Zincon Method

Required Reagents	Zinc reagent pillow  Cyclohexanone solu	ition	Cat. NO.	13610-00
Interferences	Aluminum	Above 6 mg/L		
	Cadmium	Above 0.5 mg/L		
	Copper	Above 5 mg/L		
	Iron, Ferric	Above 7 mg/L		
	Manganese	Above 5 mg/L		
	Nickel	Above 5 mg/L		
	Organic material	Large amounts may interfere. Pretreat the sample with a mild digestion.		
	Highly buffer & Extreme pH			
Sampling Storage	Collect samples in acid-cleaned plastic or glass bottles. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with nitric acid (about 2 mL per liter).			
& Preservation	Preserved samples may be stored up to six months at room temperature. Before analysis, adjust the pH to 4.5 with 5.0 N NaOH. Do not exceed pH 5 as zinc may precipitate.			
Tips & Techniques	Digestion is required for determining total zinc.			
	Zinc Reagent contains cyanide and is very poisonous if taken internally or if fumes are inhaled. Do not add to an acidic sample (pH< 4). Use only glass-stoppered cylinders in this procedure.			
	Wash glassware with 1:1 HCl and rinse with deionized water before use.			
	Use plastic droppers in this procedure. Droppers with rubber bulbs may contaminate the reagent.			
	Adjust the pH of the sample after the total phosphorus digestion to 4.5 with NaOH before analysis.			
	When Zinc Reagent pillow is dissolved, sample should be orange. If the sample is brown or blue, either the zinc concentration is too high, or an interfering metal is present. Dilute the sample and repeat the te			

Zinc

Digestion

#### Zincon Method

Digestion is required if total zinc is being determined. The following is not the USEPA digestion.

# 1. If nitric acid has not been added to the sample previously, add 5 mL of Concentrated Nitric Acid to one liter of sample (use a glass serological pipet and pipet filler). If the sample was acidified at collection, add 3 mL of nitric acid to one liter of sample.

#### Digestion

- 2. Transfer 100 mL of acidified sample to a 250-mL Erlenmeyer flask.
- 3. Add 5 mL of 1:1 Hydrochloric Acid.
- 4. Heat sample on a Hot Plate for 15 minutes at 95 °C (203 °F). Make sure the sample does not boil.
- 5. Filter cooled sample through a membrane filter and adjust the volume to 100 mL with Deionized Water
- 6. Adjust the pH to 4 ~ 5 with 5N NaOH Solution before analysis.

Zinc 96

#### Zincon Method



1. Fill a 25 mL graduated cylinder with 20mL of sample.



2. Add the contents of one Zinc Reagent Pillow to the cylinder. Stopper. Invert several times to dissolve the powder completely. Inconsistently readings may result for low zinc concentrations if all the particles are not dissolved.



3. Pour 10 mL of the solution into a sample cell.(the blank)



4. Add 0.5 mL of Cyclohexanone solution to the remaining solution in the cylinder.

A 30 second reaction period will begin.

During the reaction period, stopper the cylinder and shake vigorously.



**5.** A **30 second** reaction period will begin. Pour the solution from cylinder into a sample cell. (the prepared sample)



**6.** After choosing C-MAC mode in the program, choose **Prog.# 97.** 

(HACH DR/890 : 97

DR/2010 & 2500 : 780

DR/4000 : 3850)



**7.** Wipe the blank and place it into the cell holder.

Place the cover on the sample cell. Press Zero.



**8.** Wipe the prepared sample and place it into the cell holder.

Place the cover on the sample cell. Press Enter.

(Results will appear in mg/L Zn)

Zinc